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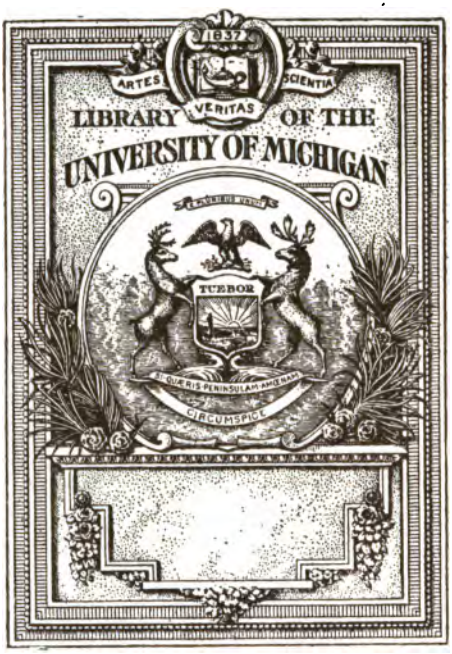

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1888 **Die Prüfung der Chemischen Reagentien auf Reinheit.** Von Dr. C. KRAUCH. Published by Julius Springer, Berlin.

1891 **Die Prüfung der Chemischen Reagentien auf Reinheit.** Von Dr. C. KRAUCH. Published by Julius Springer, Berlin.

1896 **Die Prüfung der Chemischen Reagentien auf Reinheit.** Von Dr. C. KRAUCH. Published by Julius Springer, Berlin.

1902 **The Testing of Chemical Reagents for Purity.** By Dr. C. KRAUCH. Translated by J. A. WILLIAMS, F.C.S., and L. W. DUPRÉ, with additions and emendations by the author. (Translation of 1896 Edition.) Published by Maclaren & Sons, London.

1905 **Prüfung Der Chemischen Reagenzien auf Reinheit.** Von E. MERCK. Published by E. Merck, Darmstadt.

1907 **Chemical Reagents: Their Purity and Tests.** By E. MERCK. Authorised translation by HENRY SCHENCK, A.B. (Translation of 1905 Edition.) Published by D. Van Nostrand Co., New York.

1912 **Prüfung der Chemischen Reagenzien auf Reinheit.** (Zweite Auflage.) Von E. MERCK. Published by E. Merck, Darmstadt.

1914 **Chemical Reagents: Their Purity and Tests.** By E. MERCK. Authorised translation by HENRY SCHENCK, A. B. (Translation of 1912 Edition.) Published by Merck & Co., New York. Supplied by D. Van Nostrand Co., New York.

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CHEMICAL REAGENTS

THEIR PURITY AND TESTS

AUTHORIZED TRANSLATION

OF

PRÜFUNG DER CHEMISCHEN REAGENZIEN AUF REINHEIT

(ZWEITE AUFLAGE)

VON

E. MERCK

BY

HENRY SCHENCK, A.B.(HARVARD)

SECOND EDITION, REVISED

MERCK & CO.

NEW YORK

1914

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TRANSLATOR'S PREFACE

The second edition of "Prüfung der chemischen Reagenzien auf Reinheit," presented here in translation, does not depart essentially in its method of treatment from the previous one. Since the first edition of the work appeared, numerous articles have come into prominence as reagents, however, and new uses have been found for chemicals long part of the laboratory equipment, so that, besides the introduction of entirely new subjects,* additions and changes have also been made in the text of the earlier edition. Frequently, too, these changes have materially raised the standard of purity. Coincident with this improvement is the inclusion among the references of important new contributions upon the uses and methods of testing reagent chemicals.

There have also been made several deletions: Acid Oxalic Sublimed, Indigotin, Kassner's Mixture, and a few of the less impor-

* As subjects not heretofore treated in the work but introduced for the first time in this edition, may be mentioned the following:

Alphanaphthylamine, Ammonium Chromate, Ammonium Molybdate Solution, Ammonium Persulphate, Benzidine for Blood Test, Bismuth and Potassium Iodide Solution, Carbon Tetrachloride, Carmine-Fibrin, Chromium Trioxide for Carbon Determination, Cobalt Nitrate, Free from Nickel, Cobalt Oxide, Copper Hydroxide, Copper Oxide-Asbestos, Devarda's Metal, Dicyandiamidine Sulphate, Dimethylglyoxime, Dimethylparaphenylenediamine Hydrochloride, Ferric Oxide, Glass Wool, Hydrazine Sulphate, Indigo Carmine, Indigo Solutions, Lead Peroxide Granulated, Magnesia Mixture, Magnesite, Manganese Metaphosphate Solution, Methyl Red, Platinized Pumice Stone, Poirrier's Blue C4B, Potassium Persulphate, Quartz Sand, Sea Sand, Silver-Asbestos, Sodium-Cobaltic Nitrite Solution, Tetramethylparaphenylenediamine Hydrochloride, Yellow Oxide of Mercury.

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tant dilutions of the acids have been deleted. Gallein, Dry, has taken the place of Gallein, Liquid. The tables of equivalents of standard solutions have been replaced, in response to a suggestion, by a table giving approximate strengths and brief directions for the preparation of solutions for reagent purposes, compiled from published writings.

In adapting the standards to the needs of American chemists, furthermore, it has been necessary in several cases to deviate from the German standard in the translation. Instances of such changes are the tests for tartaric acid in citric acid and in succinic acid, for silicofluoride in ammonium fluoride, for hydriodic acid in hydrobromic acid, etc.; more or less complete revision is to be found in the texts of Acid Hydrochloric, Acid Nitric, etc.

Another feature introduced in this translation is the parenthetical statement appended to each test giving in terms of percentage the minimum amount of the impurity which would be recognized by the test.* In many cases where gravimetric or volumetric determinations are made, these percentage figures are, of course, exact. In other cases, however, the qualitative tests had in a certain sense to be given a quantitative value, which had to be determined by experiments: Known quantities of an impurity were put into the chemical or its solution, and gradually added to until the given qualitative test for that impurity showed a reaction.

The figures thus obtained for qualitative tests are by no means to be considered final, as they depend to an extent on delicacy of manipulation; on the interpretation of such terms as "faint," "slight," "cloudy," etc.; and also on the absence of other impurities, the presence of which might conceivably alter them. Yet, taken by and large, they will give a more definite idea of the chemicals that conform to the tests laid down; and, furthermore, they will and do sometimes in most striking fashion show the delicacy or lack of it of some of the tests. Thus they carry a two-fold pos-

* When impurities are tested for not singly but as classes—as for instance "heavy metals," "aldehydes," etc.,—a definite percentage statement becomes obviously impossible. Also, when the text confines the test with H_2S for heavy metals to either alkaline or acid solution, the statement as to the absence of heavy metals relates necessarily to their absence in the prescribed solution only.

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sibility—that of pointing the way to improvements in both chemicals and tests.

While these are being worked out, however, the parenthetical percentage statements will, in the meantime, prove of use to the analyst when he selects reagents for particular purposes, estimates the relative value of tests, or makes allowances and corrections in his findings or in those of his assistants or students.

THE TRANSLATOR.

NEW YORK,
April 15, 1914.

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TRANSLATOR'S NOTE

For the sake of brevity, expressions throughout the book are sometimes used without full qualification. The following is then to be their interpretation:

Atomic Weights, Quantitative Calculations, etc., are based on the table of International Atomic Weights for 1913 in which O = 16.

Specific Gravities are given at +15° C. compared with water at +15° C. When a specific gravity is given with only two decimal places, it is understood that a variation in the third decimal is permissible.

Unweighable Residue or words to that effect apply to residues weighing 0.0005 gm. or less. In residue determinations when filtration is employed, ashless filters are to be used in collecting residues for ignition.

Solutions of chemicals to be tested, in the absence of specific directions for their preparation, are made from 1 gm. diluted to 20 c.c. or proportionately.

Parts by Weight are to be understood when the term "parts" is used or implied. For instance, the expression 1 : 20 means 1 gm. in 20 gm.

Volumetric Determinations can of course be made with other solutions than the potassium hydroxide and hydrochloric acid solutions generally selected, provided they be equivalent. In such substitution, however, due regard must be given to the suitableness of the indicator.

The **Boiling-point** of a substance is affected by variation in atmospheric pressure, and this fact must be taken into account when the boiling-point is employed as a test of purity.

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Nitric Acid has a specific gravity of 1.153.

Hydrochloric Acid, one of 1.124.

Ammonia Water, that of 0.96.

The **Marsh Apparatus** should be set up with a 200 c.c. generating flask.

The tests presuppose the use of Jena or some equally resistant **Glassware.**

The abbreviations of the names of periodicals in the references are those commonly used in abstracts in American journals.

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CHEMICAL REAGENTS

ACETONE

$(CH_3)_2CO$. Mol. Wt. 58.04

A clear, colorless liquid, boiling at 55° to 56° C., and having a specific gravity of 0.798.

TESTS OF PURITY

Residue on Evaporation. — 25 c.c. of acetone on evaporation should leave no weighable residue. (Indicating less than 0.0025 per cent.)

Acids. — Acetone should not redden blue litmus paper. (Indicating none present.)

Solubility in Water. — Acetone should be miscible with an equal volume of water, yielding a clear liquid.

Aldehydes. — On heating 10 c.c. of acetone with 5 c.c. of ammoniacal silver nitrate solution* (protected from the light) for fifteen minutes on a water-bath of about 50° C., the mixture must not acquire a brown color, and no metallic silver should deposit. (Indicating none present.)

Substances Oxidizable by Permanganate. — On adding one drop of a 1 : 1000 solution of potassium permanganate to 10 c.c. of acetone, and maintaining a temperature of not over 15° C., the pink color should not be entirely discharged within fifteen minutes. (Indicating none present.)

Water. — On mixing equal volumes of acetone and petroleum benzin (boiling-point 40° to 70° C.), two layers should not be formed. (Indicating less than 3 per cent.)

NOTE. — Regarding the quantitative determination of acetone, see G. Lunge, Chem.-Tech. Untersuch.-Meth., 6 ed., **3**, 114 and 924 (1911). Compare Allen's Commercial Organic Analysis, 4 ed., **1**, 100 (1909). See also G. Krämer, Ber., 13, 1000 (1880); *abst.*, J. Chem. Soc., **38**, 826 (1880). J. Messinger, Ber., **21**, 3366 (1888); *abst.*, J. Chem. Soc., **56**, 313 (1889). H. Strache, Monatsh. Chem., **12**, 524 (1891) [or Z. anal. Chem., **31**, 573 (1892)]; *abst.*, J. Chem. Soc., **62**, 546 (1892). F. Robineau and G. Rollin, Z. anal. Chem., **33**, 87 (1894); *abst.*, J. Chem. Soc., **64**, II, 556 (1893).

* The ammoniacal silver nitrate solution is made by mixing 10 c.c. of silver nitrate solution (1 : 20) with 5 c.c. of ammonia water (10 per cent).

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ACID ACETIC

$HC_2H_3O_2$. Mol. Wt. 60.03

I

ACID ACETIC, 99.5 PER CENT

A clear, colorless liquid, with a strong odor; miscible in all proportions with water or alcohol, and containing at least 99.5 per cent of $HC_2H_3O_2$. Specific gravity about 1.048 at 25° C. At about +15.65° C. it solidifies.

TESTS OF PURITY

Non-volatile Matter. — 100 c.c. of acetic acid should leave no weighable residue on evaporation and ignition. (Indicating less than 0.00048 per cent.)

Hydrochloric Acid. — 5 c.c. of acetic acid diluted with 50 c.c. of water should not be changed on the addition of 5 c.c. of nitric acid followed by silver nitrate solution. (Indicating less than 0.0003 per cent Cl.)

Sulphuric Acid. — Add to 100 c.c. of acetic acid a small amount of sodium carbonate, evaporate the solution to dryness, take up the residue with 50 c.c. of water, and add barium chloride solution. No weighable precipitate of barium sulphate should form on standing fifteen hours. (Indicating less than 0.00001 per cent SO_3.)

Heavy Metals. — (*a*) A mixture of 20 c.c. of acetic acid and 100 c.c. of water should not be affected by passing through it a current of hydrogen sulphide gas. (Indicating none present.)

(*b*) Evaporate 100 c.c. of the acid to a small volume, add a slight excess of ammonia water, and dilute with water to a 100 c.c. A green or dark color should not develop on adding a few drops of ammonium sulphide solution. (Indicating none present.)

Iron. — 12.5 c.c. of the acid diluted with an equal volume of water and boiled after adding a few drops of nitric acid, should not develop a red color on addition of potassium sulphocyanate solution. (Indicating less than 0.0001 per cent Fe.)

Formic and Sulphurous Acids. — 2 c.c. of the acid are just neutralized with ammonia water and a little silver nitrate solution is added. No dark color or deposit should appear on boiling two minutes. (Indicating less than 0.05 per cent SO_2, or less than 0.4 per cent HCOOH.)

Substances Reducing Permanganate. — 2 c.c. of the acid are diluted with 10 c.c. of water and 0.1 c.c. of decinormal solution of potassium permanganate added. The pink color should not be entirely changed to brown within two hours. (Indicating none present.)

Other Empyreumatic Bodies. — No smoky color should be noticeable upon making 10 c.c. of the acid alkaline with solution of potassium hydroxide. (Indicating none present.)

Earths. — 10 c.c. of acetic acid diluted with 100 c.c. of water and treated with an excess of ammonia water, should not become turbid on adding ammonium oxalate solution. (Indicating less than 0.003 per cent earths as Ca.)

Quantitative Determinations. — Dilute 5 gm. of acetic acid with sufficient water to make 50 c.c. Titrate 10 c.c. of this solution with normal potassium hydroxide solution, using phenolphthalein as the indicator. At least 16.5 c.c.

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of the normal potassium hydroxide solution should be required to produce the pink color.

1 c.c. of normal KOH = 0.06003 gm. $HC_2H_3O_2$, log. 77837.

The acetic acid content may also be ascertained from the congealing-point,* or from the specific gravity if due note be taken of the fact that the specific gravities above 1.0553 represent in each case two liquids of different acetic acid content.†

II

ACID ACETIC, GLACIAL, 96 PER CENT

A clear, colorless liquid with a strong odor; congealing at about 10° C., having a specific gravity of at most 1.064 and boiling at 110°–119° C. It contains at least 96 per cent of $HC_2H_3O_2$.

TESTS OF PURITY

The tests of purity and the quantitative determination are to be carried out as described under Acid Acetic, 99.5 per cent, observing the conditions there given.

III

ACID ACETIC, 90 PER CENT

A colorless liquid having a specific gravity 1.071 and containing at least 90 per cent of $HC_2H_3O_2$.

TESTS OF PURITY

The tests to be made are those given under Acid Acetic, 99.5 per cent, observing the conditions there described.

IV

ACID ACETIC, 36 PER CENT

A clear, colorless liquid, specific gravity about 1.048, and containing about 36 per cent of $HC_2H_3O_2$.

TESTS OF PURITY

The tests of purity and details of execution are the same as those given under Acid Acetic, 99.5 per cent. But instead of 1 gm. (or c.c.) of 99.5 per cent, use 2.5 gm. (or c.c.) of the 36 per cent acid.

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* See Lunge, Chem.-Tech. Untersuch.-Meth., 5 ed., **3**, 934 (1905). Compare Landolt-Börnstein-Roth, Physikalisch-Chemische Tabellen, 4 ed., 296 (1912).

† Compare Lunge, Chem.-Tech. Utersuch.-Meth., 6 ed., **3**, 954 (1911). Also U. S. Pharmacopœia, VIII, p. 610 (1905). Landolt-Börnstein-Roth, loc. cit.

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ACID ACETIC, DILUTED, 30 PER CENT

A colorless liquid having the specific gravity 1.040–1.042. The liquid contains about 30 per cent of $HC_2H_3O_2$.

TESTS OF PURITY

The tests to be made are those given under Acid Acetic, 99.5 per cent, observing the conditions there described. But instead of 1 gm. or c.c. of the 99.5 per cent, use 3 gm. or c.c. of the 30 per cent acid.

(ACID) ACETIC ANHYDRIDE

$(CH_3CO)_2O$. Mol. Wt. 102.05.

A colorless liquid of pungent odor, boiling at about 137° C. Specific gravity 1.080 to 1.085.

On pouring acetic anhydride into water, it does not at first mix with the latter, but sinks to the bottom of the vessel. Gradually, however, it dissolves, forming acetic acid.

TESTS OF PURITY

Hydrochloric Acid. — 1 c.c. of acetic anhydride dissolved in 50 c.c. of water, and acidulated with 5 c.c. of nitric acid (sp.gr. 1.15), should show at most a slight opalescence with silver nitrate solution. (Indicating less than 0.002 per cent Cl.)

Non-volatile Matter. — 10 c.c. of acetic anhydride should leave no weighable residue on evaporation. (Indicating less than 0.005 per cent.)

Quantitative Determination. — Dissolve 5 gm. of acetic anhydride in water and dilute to 50 c.c. Titrate 10 c.c. of this solution with normal potassium hydroxide solution, using phenolphthalein as indicator. It should require at least 19.3 c.c. of the normal potassium hydroxide solution to develop the pink color.

1 c.c. of normal KOH = 0.051025 gm. of $(CH_3CO)_2O$, log. 70778.

(ACID) BORIC ANHYDRIDE

(Boron Trioxide; Boric Acid, Fused)

B_2O_3. Mol. Wt. 70.0.

Brittle, vitreous, hygroscopic lumps. Used in silicate analyses, according to Jannasch.*

TESTS OF PURITY

Silica, Alkalies, etc. — Add 50 c.c. of methyl alcohol-hydrochloric acid† to

* References: P. Jannasch and O. Heidenreich, Ztschr. anorgan. Chem., **12**, 211 (1896) [or Ztschr. anal. Chem., **36**, 382 (1897)]; *abst.*, J. Chem. Soc., **70**, II, 576 (1896).

† The methyl alcohol-hydrochloric acid is prepared by saturating pure, anhydrous methyl alcohol, keeping the temperature low, with thoroughly dried

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5 gm. of the powdered boric anhydride* in a weighed platinum dish, stir with a platinum wire until completely dissolved, and evaporate the liquid over a small flame, the platinum dish being placed on wire gauze. If a residue remains, it is treated anew with 25 c.c. of the methyl alcohol-hydrochloric acid, the liquid evaporated, and the dish and contents gently ignited. There must remain no weighable residue.† (Indicating less than 0.01 per cent.)

ACID CARMINIC ‡

$C_{22}H_{22}O_{13}$. Mol. Wt. 494.16

Purple-brown, amorphous masses, affording a dark red powder on trituration. Carminic acid is easily soluble in water and in alcohol, but is insoluble in benzene and chloroform. It has no constant melting-point.

TESTS OF PURITY

Solubility. — 1 gm. of carminic acid dissolves completely in 2 c.c. of water. The addition of 20 c.c. of 90 per cent alcohol to this solution should cause no appreciable precipitation.

If the carminic acid is intended to be used as an indicator in acidimetry, it must first be tested as to its sensitiveness, as follows:

Dissolve 1 gm. of the carminic acid in 100 c.c. of water and add one drop of this solution to a solution of 5 gm. of ammonium chloride in 50 c.c. of water. The addition to this latter solution of one drop of decinormal potassium hydroxide solution should cause a change in color from yellowish-red to violet-red.

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hydrochloric acid gas (prepared most readily by dropping concentrated sulphuric acid into concentrated hydrochloric acid). On standing for some time, however, the methyl alcohol-hydrochloric acid loses its original effectiveness.

* The powdering must be done in an agate mortar, since the rubbing up of hard pieces of the anhydride in porcelain or other mortars is apt to introduce impurities into the chemical.

† If a residue remains after the second treatment with methyl alcohol-hydrochloric acid and ignition, repeat the entire operation a third time in order to make sure it is not some of the anhydride still unvolatilized.

‡ References: E. Schunck and L. Marchlewski, Ber., **27**, 2979 (1894); *abst.*, J. Chem. Soc., **68**, I, 67 (1895). C. Liebermann and H. Voswinckel, Ber., **30**, 688 (1897); *abst.*, J. Chem. Soc., **72**, I, 292 (1897). Ber., **30**, 1731 (1897); *abst.*, J. Chem. Soc., **72**, I, 539 (1897). C. Liebermann, P. Höring, and F. Wiedemann, Ber., **33**, 149 (1900); *abst.*, J. Chem. Soc., **78**, I, 236 (1900). J. Landau, Ber., **33**, 2446 (1900); *abst.*, J. Chem. Soc., **78**, 661 (1900); C. Liebermann and J. Landau, Ber., **34**, 2153 (1901); *abst.*, J. Chem. Soc., **80**, I, 545 (1901). C. Liebermann and S. Lindenbaum, Ber., **35**, 2910 (1902); *abst.*, J. Chem. Soc., **82**, I, 787 (1902). O. Dimroth, Ber., **42**, 1611, 1735 (1909); *abst.*, J. Chem. Soc., **96**, I, 485 (1909).

Regarding the use of carminic acid as a reagent see T. J. Bogomoloff and N. J. Wassilieff, Petersburger med. Wochschr., 294 (1897); *abst.*, E. Merck, Annual Report, **27** (on year 1898). Compare Pharm. Zentralhalle, **40**, 120 (1899).

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ACID CITRIC

$C_6H_8O_7+H_2O$. Mol. Wt. 210.08

Colorless, odorless, rhombic prisms, efflorescing superficially in warm air.

Citric acid is soluble in 0.75 part of cold and in 0.5 part of boiling water; in 1 part of alcohol (about 85 per cent by weight), and in 50 parts of ether.

TESTS OF PURITY

Oxalic Acid. — On dissolving 10 gm. of citric acid in 20 c.c. of water, and adding 5 c.c. of a 1 : 2 potassium acetate solution and 50 c.c. of 85 per cent alcohol, no turbidity should be produced within two hours. (Indicating less than 2.8 per cent $H_2C_2O_4$.)

Tartaric Acid* and Sugar. — 1 gm. of citric acid and 10 c.c. of sulphuric acid (sp.gr. 1.84) are triturated in a porcelain mortar previously rinsed with sulphuric acid. When this mixture is then heated in a test-tube for an hour in a boiling water-bath it acquires at most a slight yellow color, but no brown color should develop. (Indicating less than 1 per cent $C_4H_6O_6$, and less than 0.05 per cent saccharose.)

Sulphuric Acid. — 20 c.c. of the 1 : 10 aqueous solution must show no change on adding barium chloride solution and allowing to stand fifteen hours. (Indicating less than 0.002 per cent SO_3.)

Calcium. — 20 c.c. of the 1 : 10 aqueous solution must show no change on the addition of ammonium oxalate solution. (Indicating less than 0.01 per cent Ca.)

Lead.† — A solution of 5 gm. of citric acid in 10 c.c. of water, to which 12 c.c. of ammonia water are added, should not acquire a dark color upon adding hydrogen sulphide water. (Indicating less than 0.001 per cent Pb.)

Non-volatile Matter. — 1 gm. of citric acid must leave no weighable residue on ignition. (Indicating less than 0.05 per cent.)

Quantitative Determination. — On dissolving 1 gm. of citric acid in 30 c.c. of water and titrating with normal potassium hydroxide solution, using phenolphthalein as indicator, not less than 14.2 c.c. of the alkali solution should be required to develop the pink color.

1 c.c. of normal KOH = 0.07002 gm. of $C_6H_8O_7+H_2O$, log. 84522.

ACID GALLIC

$C_7H_6O_5+H_2O$. Mol. Wt. 188.06.

Colorless or slightly yellowish needles or prisms, which melt and slowly decompose at 220° C. Gallic acid dissolves in 85 parts

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* For detection of tartaric acid in citric acid see Cailletet, Arch. Pharm. (3), **13**, 468 (1878); *abst.* Chem. Zentrbl. (3) **10**, 14, (1879); *abst.*, J. Chem. Soc., **35**, 674 (1879). M. L. Crismer, Bull. Soc. Chim. Paris, (3), **6**, 23 (1891); *abst.*, J. Chem. Soc., **62**, 546 (1892). Salzer, Chem. Zentr. (3), **19**, 1244 (1888); *abst.*, J. Chem. Soc., **54**, 996 (1888). O. Spindler, Chem. Ztg., **28**, 15 (1904); *abst.* Chem. Zentr. (5), 8, 696 (1904); *abst.*, J. Chem. Soc., **86**, II, 152 (1904).

† Regarding the examination of citric acid for lead compounds see M. Bucket, Z. anal. Chem., **32**, 465 (1893); *abst.*, J. Chem. Soc., **64**, II, 557 (1893). Tatlock and Thompson, Analyst, **33**, 173 (1908); *abst.*, C. A., **2**, 2269 (1908).

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of cold water, in 3 parts of boiling water, and in about 6 parts of 95 per cent alcohol. In ether* (sp.gr. 0.72) it is difficultly soluble.

TESTS OF PURITY

Solubility in Water. — 1 gm. of gallic acid must dissolve completely in 20 c.c. of water on heating. The solution must be colorless or only very faintly yellowish.

Water Content. — On drying 1 gm. of gallic acid at 100° C. to constant weight, it should not lose more than 0.10 gm. in weight. (Indicating not more than 10 per cent.)

Sulphuric Acid. — On adding 1 c.c. of hydrochloric acid and some barium chloride solution to a hot 1 : 50 aqueous solution of gallic acid, a precipitate of barium sulphate must not be visible after standing one hour at about 50° C. (Indicating less than 0.005 per cent SO_3.)

Inorganic Matter. — 1 gm. of gallic acid must leave no weighable residue on ignition. (Indicating less than 0.05 per cent.)

ACID HYDRIODIC

HI. Mol. Wt. 127.93

I

ACID HYDRIODIC, Sp. Gr. 1.50

A clear, colorless liquid which, on exposure to light and air, rapidly becomes yellow to brown, due to the separation of iodine. Specific gravity 1.50. The liquid contains about 43 per cent of HI.

TESTS OF PURITY

Non-volatile Matter and Phosphorus. — 5 gm. of hydriodic acid should not leave more than 0.002 gm. residue on evaporation. Upon gentle ignition the residue should not burn (indicating no phosphorus present), and after the ignition no weighable residue should remain. (Indicating less than 0.01 per cent non-volatile matter.)

Sulphuric Acid. — On diluting 3 c.c. of hydriodic acid with 50 c.c. of water and adding barium chloride solution, no barium sulphate should precipitate on standing fifteen hours. (Indicating less than 0.01 per cent SO_3.)

Heavy Metals and Earths. — On diluting 10 c.c. of hydriodic acid with 100 c.c. of water and passing hydrogen sulphide gas into it no dark-colored precipitate should form; then after adding an excess of ammonia water and some ammonium sulphide solution neither a green color nor a precipitate should be visible. (Indicating no heavy metals present.) Three c.c. of the hydriodic acid diluted with

* The solubility of gallic acid in ether depends very largely on the amount of alcohol in the ether.

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30 c.c. of water and made alkaline with ammonia water should show no change upon the addition of ammonium oxalate solution. (Indicating less than 0.005 per cent earths as Ca.)

Hydrochloric and Hydrobromic Acids. — Dilute 1 gm. of hydriodic acid with 20 c.c. of water, add an excess of silver nitrate solution followed by 30 c.c. of ammonia water, shake well, and filter. On acidulating the filtrate with nitric acid, only a slight turbidity may develop, never a precipitate.* (Indicating less than 0.002 per cent as Cl.)

Quantitative Determination. — Dilute 5 gm. of hydriodic acid with 50 c.c. of water and titrate with normal potassium hydroxide solution, using methyl orange as an indicator. At least 17 c.c. of the normal alkali solution must be required to effect the change in color.

1 c.c. of normal KOH = 0.12793 gm. of HI, log. 10697.

II

ACID HYDRIODIC, Sp. Gr. 1.70

A yellowish or brownish liquid of specific gravity 1.70. The acid contains about 57 per cent of HI, and is used for methoxyl determination, according to Zeisel.

TESTS OF PURITY

Tests for non-volatile impurities and sulphuric acid are to be made, observing the conditions given under Acid Hydriodic, Sp.Gr. 1.50. In titrating this acid, phenolphthalein is used as the indicator.

ACID HYDROBROMIC

HBr. Mol. Wt. 80.93

A clear, colorless, or faintly yellowish liquid, of specific gravity 1.38. It contains about 40 per cent of HBr.

TESTS OF PURITY

Non-volatile Matter. — 10 gm. of hydrobromic acid on evaporation on the water-bath should leave not more than 0.001 gm. of residue. (Indicating not more than 0.01 per cent.)

Arsenic. — A mixture of 1 c.c. of hydrobromic acid and 3 c.c. of stannous chloride solution should show no dark color in one hour. (Indicating less than 0.0015 per cent As.)

Sulphuric Acid. — On diluting 5 c.c. of hydrobromic acid with 50 c.c. of water and adding barium chloride solution, a precipitate of barium sulphate should not form on standing fifteen hours. (Indicating less than 0.0075 per cent SO_3.)

* A slight turbidity is permitted, because silver iodide is not absolutely insoluble in ammonia water.

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Heavy Metals. — (*a*) Dilute 10 c.c. of hydrobromic acid with 40 c.c. of water and introduce hydrogen sulphide gas. No change should appear. (Indicating none present.)

(*b*) Dilute 10 c.c. of hydrobromic acid with 10 c.c. of water, and add 15 c.c. of ammonia water, followed by a few drops of ammonium sulphide solution. Neither a brown coloration nor a precipitate should appear. (Indicating none present.)

(*c*) Dilute 5 c.c. of hydrobromic acid with 25 c.c. of water and add potassium sulphocyanate solution. No reddish coloration should occur. (Indicating less than 0.00015 per cent Fe.)

Hydrochloric Acid. — 0.2 c.c. of hydrobromic acid are diluted with 4 c.c. of water, and then mixed with 15 c.c. of decinormal silver nitrate solution. To this mixture add 6 c.c. of ammonium carbonate solution (1 part of ammonium carbonate, 1 part of ammonia water, and 3 parts of water), shake for five minutes, and filter. The filtrate acidulated with nitric acid may exhibit at most a faint turbidity. (Indicating less than 0.06 per cent HCl.)

Hydriodic Acid. — On adding 1 drop of ferric chloride solution to 5 c.c. of hydrobromic acid, and shaking with 5 c.c. of chloroform, the latter should not acquire a violet color. (Indicating less than 0.0156 per cent HI.)

Phosphorous and Phosphoric Acids. — Heat 1 c.c. of hydrobromic acid with 1 c.c. of nitric acid to boiling, allow to cool, and then add 4 c.c. of ammonia water followed by 5 c.c. of magnesia mixture.* No precipitate should form on standing fifteen hours. (Indicating less than 0.0075 per cent P_2O_5.)

Calcium. — To 10 c.c. of hydrobromic acid diluted with 10 c.c. of water and mixed with 15 c.c. of ammonia water, ammonium oxalate solution is added. No precipitate should form on standing two hours. (Indicating less than 0.005 per cent Ca.)

Quantitative Determination. — Dilute 5 gm. of hydrobromic acid with 50 c.c. of water, and titrate with normal potassium hydroxide solution, using methyl orange as indicator. At least 24.6 c.c. of the normal alkali solution should be required to effect the change in color.

1 c.c. of normal KOH = 0.08093 gm. of HBr, log. 90806.

ACID HYDROCHLORIC

HCl. Mol. Wt. 36.47

I

ACID HYDROCHLORIC, Sp.Gr. 1.19

(Fuming Hydrochloric Acid)

A clear, colorless liquid, fuming in the air, and of specific gravity 1.19. The liquid contains about 37 per cent of HCl.

TESTS OF PURITY

Sulphuric Acid. — Evaporate 85 c.c. of hydrochloric acid in a platinum dish to about 5 c.c., dilute the residue with 20 c.c. of water, and add barium chloride

* Dissolve 110 gm. crystallized magnesium chloride and 140 gm. ammonium chloride in 1300 gm. of water, then add 700 gm. ammonia water.

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solution. No precipitate of barium sulphate should form on standing fifteen hours. (Indicating less than 0.000125 per cent SO_3.)

Non-volatile Matter. — 85 c.c. of hydrochloric acid, when evaporated in a platinum dish on the water-bath, and subsequently ignited, must leave no weighable residue. (Indicating less than 0.0005 per cent.)

Chlorine. — Dilute 10 c.c. of hydrochloric acid with 50 c.c. of water and add zinc iodide-starch solution. The liquid must not develop a blue color within ten minutes. (Indicating less than 0.00016 per cent Cl.)

Sulphurous Acid. — 50 c.c. of water are colored blue by the addition of 1 drop of decinormal iodine solution and a few drops of starch solution. Then add a mixture containing 5 c.c. of the hydrochloric acid to be tested and 50 c.c. of water. The liquid must not be decolorized after being shaken. (Indicating less than 0.005 per cent as SO_2.)

Heavy Metals. — (*a*) Dilute 20 c.c. of hydrochloric acid with 200 c.c. of water, warm the liquid to about 70° C., and pass into it hydrogen sulphide gas. No change should be apparent. (Indicating none present.)

(*b*) Evaporate 60 c.c. of the acid to about 10 c.c., dilute with water to 40 c.c., add 50 c.c. ammonia water and a few drops of ammonium sulphide solution. No green or dark color should develop, nor should a precipitate form. (Indicating none present.)

(*c*) A reddish color should not develop on adding potassium sulphocyanate solution to 6 c.c. of the acid diluted with water to 20 c.c. (Indicating less than 0.0001 per cent Fe.)

Calcium. — On diluting 20 c.c. of hydrochloric acid with 20 c.c. of water and adding 50 c.c. of ammonia water, followed by a few c.c. of ammonium oxalate solution, no precipitate should form on standing two hours. (Indicating less than 0.001 per cent Ca.)

Arsenic. — 100 c.c. hydrochloric acid are added by drops to 30 c.c. of nitric acid (sp.gr. 1.3) heated to about 60° C. After the addition of 5 c.c. of arsenic-free sulphuric acid (sp.gr. 1.84) to the mixture it is evaporated as far as possible on a water-bath, and then heated on a sand-bath until it gives off fumes of sulphuric trioxide. A Marsh apparatus is started, using 20 gm. of arsenic-free, granulated zinc and dilute (1 : 5) sulphuric acid; then the residue from the above evaporation is washed into the generating flask of the apparatus, diluted with 20 c.c. of water. After the apparatus has been in action for one hour, there may be at most a very slight deposit in the reduction tube, but no distinctly visible arsenic mirror. (Indicating not more than 0.000005 per cent As.)

Quantitative Determination. — The strength of the acid may be most simply ascertained from the specific gravity.* On diluting 3 gm. of hydrochloric acid with 50 c.c. of water, and titrating with normal potassium hydroxide solution, using methyl orange as indicator, not less than 30.5 c.c. of the normal alkali should be required to effect the color change.

1 c.c. of normal KOH = 0.03647 gm. of HCl, log. 56194.

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*See the table by Lunge and Marchlewski, in Lunge's Chem.-Tech. Untersuch.-Meth., 5 ed., I, 419 (1904); U. S. Pharmacopœia, VIII, p. 612 (1905). Compare J. Soc. Chem., Ind., 24, 789 (1905); also Landolt-Börnstein-Roth, Physikalish-Chemische Tabellen, 4 ed., 262 (1912).

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ACID HYDROCHLORIC, Sp.Gr. 1.126

II

(Diluted Hydrochloric Acid)

A clear, colorless liquid, of specific gravity 1.126, and containing about 25 per cent of HCl.

TESTS OF PURITY

The tests to be made are those given under Fuming Hydrochloric Acid. But, for every 10 gm. (or c.c.) of fuming acid, use 15 gm. (or c.c.) of the acid of sp.gr. 1.126.

Quantitative Determination.* — Dilute 5 gm. of the acid with 50 c.c. of water, and titrate with normal potassium hydroxide solution, using methyl orange as indicator. At least 34.3 c.c. of the normal alkali should be required for neutralization.

ACID HYDROFLUORIC

HF. Mol. Wt. 20.01

A colorless, or almost colorless, liquid, containing about 48 per cent of HF. It fumes in the air.

TESTS OF PURITY

In these tests use platinum containers as much as possible.

Non-volatile Matter. — 20 gm. of hydrofluoric acid evaporated in a platinum dish, and gently ignited, should leave a residue weighing not more than 0.001 gm. (Indicating not more than 0.005 per cent.)

Sulphuric Acid. — Evaporate 2 gm. of hydrofluoric acid in a platinum dish on the water-bath, take up the residue with 10 c.c. of water, and to this solution add a few drops of nitric acid and some barium nitrate solution. An immediate turbidity must not occur; and even after some time the liquid should exhibit, at most, a slight opalescence. (Indicating less than 0.005 per cent SO_3.)

Calcium. — Dilute 5 gm. of hydrofluoric acid with 50 c.c. of water and add ammonia water in excess, followed by ammonium oxalate solution. An immediate turbidity should not occur. (Indicating less than 0.005 per cent Ca.)

Magnesium. — Dilute 5 gm. of hydrofluoric acid with 50 c.c. of water, and add ammonia water until the liquid has an alkaline reaction; on now adding ammonium phosphate solution, no precipitate should form on standing three hours. (Indicating less than 0.001 per cent Mg.)

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* See the table by Lunge and Marchlewski, in Lunge's Chem.-Tech. Untersuch.-Meth., 5 ed., I, 419 (1904); U. S. Pharmacopœia, VIII, p. 612 (1905). Compare J. Soc. Chem., Ind., **24**, 789 (1905); also Landolt-Börnstein-Roth, Physikalisch-Chemische Tabellen, 4 ed., 262 (1912).

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Heavy Metals. — (*a*) Dilute 10 gm. of hydrofluoric acid with 40 c.c. of water, warm the liquid to about 70° C. and pass through it hydrogen sulphide gas. Neither a yellow nor a dark-colored precipitate should form. (Indicating none present.)

(*b*) Dilute 5 gm. of hydrofluoric acid with 50 c.c. of water, make the solution alkaline with ammonia water, and add ammonium sulphide solution. A green coloration must not develop, nor should a precipitate form. (Indicating none present.)

Hydrochloric Acid. — Dilute 2 gm. of hydrofluoric acid with 50 c.c. of water, and add a few drops of nitric acid and silver nitrate solution. The liquid may exhibit, at most, a faint opalescence. (Indicating less than 0.001 per cent Cl.)

Hydrosilicofluoric Acid. — On diluting 5 gm. of hydrofluoric acid with 20 c.c. of water and adding 2 c.c. of a cold, saturated potassium chloride solution, the further addition of 40 c.c. of alcohol (of about 85 per cent by weight) should cause no turbidity or precipitate. (Indicating less than 0.3 per cent H_2SiF_6.)

Sulphurous Acid. — A mixture of 10 gm. of hydrofluoric acid and 40 c.c. of water should show a blue color upon the addition of one drop of decinormal iodine solution and starch solution. (Indicating less than 0.003 per cent SO_3.)

Quantitative Determination. — Dilute 2 gm. of hydrofluoric acid with 50 c.c. of water and titrate with normal potassium hydroxide solution, using phenolphthalein as indicator.

1 c.c. of normal KOH = 0.02001 gm. of HF, log. 30125.

NOTE. — Regarding the titration of hydrofluoric acid in the presence of hydrosilicofluoric acid, see Katz, Chem. Ztg., **28**, 356, 387 (1904); *abst.*, J. Chem. Soc., **86**, II, 442 (1904).

ACID HYDROSILICOFLUORIC

(Fluosilicic or Silicofluoric Acid)

H_2SiF_6. Mol. Wt. 144.32

A clear, colorless liquid having a specific gravity of about 1.06. This specific gravity corresponds to a content of about 8 per cent H_2SiF_6.

TESTS OF PURITY

Non-volatile Matter. — 5 c.c. of hydrosilicofluoric acid evaporated in a platinum dish should leave no weighable residue. (Indicating less than 0.01 per cent.)

Heavy Metals. — Dilute 5 c.c. of hydrosilicofluoric acid with 10 c.c. of water, add a few drops of hydrochloric acid and 10 c.c. of hydrogen sulphide water. No visible change should take place. (Indicating none present.)

Sulphuric Acid. — On diluting 5 c.c. of hydrosilicofluoric acid with 10 c.c. of water and adding 2 c.c. of a 10 per cent solution of barium-free strontium nitrate, no precipitate should form, even on standing fifteen hours. (Indicating less than 0.028 per cent SO_3.)

Quantitative Determination. — Titrate 10 gm. of the hydrosilicofluoric acid with normal sodium hydroxide solution, using methyl orange as indicator. Then add to the above solution (neutral to methyl orange) 50 c.c. of water, heat to boiling, and with the same alkali titrate again, using phenolphthalein as indi-

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cator. To obtain the permanent pink color should require exactly twice as much alkali as was required to make the solution neutral to methyl orange. The amount of alkali consumed in the second titration is used to calculate the strength of the acid.

1 c.c. normal NaOH = 0.03608 gm. of H_2SiF_6, log. 55727.

NOTE. — Regarding the determination of hydrosilicofluoric acid, see Katz, Chem. Ztg., **28**, 356, 387 (1904); *abst.*, J. Chem. Soc., **86**, II, 442 (1904). S. Kohn and L. Wesseley, Chem. Ztg., **31**, 204, 1207 (1907); *abst.*, J. Chem. Soc., **92**, II, 300 (1907).

ACID IODIC

HIO_3. Mol. Wt. 175.93

Colorless, rhombic crystals, or white, crystalline powder, easily soluble (1 : 1) in water, but difficultly soluble in alcohol. The aqueous solution first reddens blue litmus paper and then bleaches it.

TESTS OF PURITY

Non-volatile Matter. — On heating 2 gm. of iodic acid no weighable residue should remain. (Indicating less than 0.025 per cent.)

Solubility. — 1 gm. of iodic acid should dissolve completely in 1 c.c. of water, and yield a colorless solution.

Quantitative Determination. — Dissolve 1 gm. of iodic acid in water and dilute to 100 c.c. Dilute 10 c.c. of this solution with 50 c.c. of water, and add 2 gm. of potassium iodide and 5 c.c. of dilute sulphuric acid. Titrate the liberated iodine with decinormal sodium thiosulphate solution, using starch solution as indicator.

1 c.c. of decinormal $Na_2S_2O_3$ = 0.002932 gm. of HIO_3, log. 46716.

(ACID) IODIC ANHYDRIDE

(Iodine Pentoxide)

I_2O_5. Mol. Wt. 333.84

A white, crystalline powder, soluble in water with the formation of the hydrated acid, HIO_3, but insoluble in absolute alcohol and ether.

TESTS OF PURITY

The tests to be made are those given under Acid Iodic, and the conditions there described are to be observed.

1 c.c. of decinormal $Na_2S_2O_3$ = 0.002782 gm. of I_2O_5, log. 44436.

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ACID MOLYBDIC

H_2MoO_4. Mol. Wt. 162.02

A white or slightly yellowish powder, containing about 85 per cent of MoO_3.

TESTS OF PURITY

Solubility in Ammonia Water; Heavy Metals. — 2 gm. of molybdic acid should completely dissolve in 15 c.c. of ammonia water, yielding a clear solution. On the addition of hydrogen sulphide water to this liquid it acquires a greenish-yellow color; a brown color or a precipitate should not, however, develop. (Indicating no heavy metals present.)

Phosphoric Acid. Quantitative Determination. — As detailed under (Acid) Molybdic Anhydride, below.

(ACID) MOLYBDIC ANHYDRIDE

(Molybdic Acid, 100 per cent)

MoO_3. Mol. Wt. 144.00

A slightly yellowish powder, often exhibiting a faintly bluish tint, due to the presence of other oxides of molybdenum. The preparation contains about 100 per cent of MoO_3, and is free from ammonia and nitric acid.

TESTS OF PURITY

Alkalies. — On heating 1 gm. of molybdic anhydride in a test-tube, the particles in contact with the heated wall of the tube begin to melt at a red heat, while upon the cooler upper surface a crystalline formation of sublimed anhydride may be observed. The whole should not fuse to a dark-colored mass at a lower temperature. (Indicating no alkalies present.)

Ammonium Salts. — On boiling 1 gm. of molybdic anhydride with sodium hydroxide solution, there must be no liberation of ammonia (to be ascertained by means of moistened litmus paper). (Indicating less than 0.0035 per cent NH_3.)

Solubility in Ammonia Water; Heavy Metals. — 5 gm. of molybdic anhydride, when gently heated with 40 c.c. of ammonia water, should entirely dissolve, yielding a clear solution. The solution acquires a slight yellow color on adding hydrogen sulphide water. This color must not change within ten minutes, nor should a precipitate form. (Indicating no heavy metals present.)

Phosphoric Acid. — Dissolve 10 gm. of molybdic anhydride in 25 c.c. of ammonia water. The solution is poured into 150 c.c. of nitric acid and allowed to stand for fifteen hours at a temperature of about 40° C. A yellow precipitate must not appear. (Indicating less than 0.0005 per cent P_2O_5.)

Nitric Acid. — Shake 1 gm. of molybdic anhydride with 10 c.c. of water, and add a small crystal of sodium chloride, followed by one drop of a 1 : 1000 solution of indigo; the blue color of the solution must not disappear on adding 10 c.c. of concentrated sulphuric acid and stirring. (Indicating less than 0.0032 per cent N_2O_5.)

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Sulphates and Chlorides. — Dissolve 10 gm. of molybdic anhydride in 25 c.c. of ammonia water, pour the solution into 150 c.c. of nitric acid, and add barium nitrate and silver nitrate solutions. No turbidity should be produced. (Indicating less than 1.0 per cent SO_3, and less than 0.002 per cent Cl.)

Quantitative Determination. — Dissolve 0.5 gm. of molybdic anhydride in a mixture of 50 c.c. of water and 3 c.c. of ammonia water, with the aid of a gentle heat. Acidulate the solution with 5 c.c. of acetic acid (sp.gr. 1.040–1.042), dilute with 200 c.c. of water, heat to boiling, then add a solution of 1.5 gm. crystallized lead acetate in 20 c.c. of water. Boil for several minutes with constant stirring, whereby the precipitate, at first milky, is rendered granular and easy to filter. Collect the precipitate on a filter previously dried at 100° C. and weighed, and wash it with boiling water until the washings cease to afford a reaction with hydrogen sulphide water. Dry the precipitate to constant weight at 100° C. and then ignite a portion of it. The ignited residue has the composition $PbMoO_4$.

$PbMoO_4 \times 0.39247 = MoO_3$, log. 59380.

ACID NAPHTHYLAMINESULPHONIC

(Alphanaphthylaminesulphonic Acid; Naphthionic Acid)

$2C_{10}H_6NH_2SO_3H$ 1 : $4+H_2O$. **Mol. Wt. 464.32**

A white powder, or small, lustrous, colorless needles (when crystallized from hot water), which carbonize but do not melt on being heated. Naphthylaminesulphonic acid is soluble in about 4000 parts of cold water, more readily in hot water, scarcely soluble in alcohol, and almost insoluble in ether. The solution in ammonia water exhibits a violet fluorescence.

NOTE. — Regarding the use of naphthylaminesulphonic acid for the detection and colorimetric determination of small quantities of nitrous acid, see E. Riegler, Z. anal. Chem., **35**, 677 (1896); *abst.*, J. Chem. Soc., **72**, II, 230 (1897); Z. anal. Chem., **36**, 306 (1897); *abst.*, J. Chem. Soc., **72**, II, 385 (1897).

ACID NITRIC

HNO_3. Mol. Wt. 63.02

I

ACID NITRIC, Sp.Gr. 1.40–1.42*

A clear, colorless, or yellowish liquid, specific gravity 1.40 to 1.42, containing about 65 to 69 per cent by weight of HNO_3.

* See the tables of specific gravity and per cent in Lunge's Chem.-Tech. Untersuch.-Meth., 5 ed., I, 326 (1904); U. S. Pharmacopœia, VIII, p. 613 (1905); Landolt-Börnstein-Roth, Physikalisch-Chemische Tabellen, 4 ed., 263 (1912). Compare J. Soc. Chem., Ind., **24**, 788 (1905).

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TESTS OF PURITY

Non-volatile Matter. — 75 c.c. of nitric acid on evaporation should leave no weighable residue. (Indicating less than 0.00047 per cent.)

Sulphuric Acid. — Add to 25 c.c. of nitric acid a small amount of sodium carbonate, evaporate the solution to dryness, take up the residue with 50 c.c. of water, and add barium chloride solution. No weighable precipitate of barium sulphate should form on standing fifteen hours. (Indicating less than 0.00005 per cent SO_3.)

Hydrogen-Halogen Acids. — 50 c.c. of nitric acid diluted with 50 c.c. of water must not appear changed on the addition of silver nitrate solution. (Indicating less than 0.000015 per cent Cl.)

Heavy Metals. — Add to 25 c.c. of the acid a slight excess of ammonia water, and dilute to about 100 c.c. On adding a few drops of ammonium sulphide solution no green or dark color should develop, nor should a precipitate form. (Indicating none present.)

Iodic Acid and Iodine. — On diluting 20 c.c. of nitric acid with 20 c.c. of water, adding a small piece of metallic zinc, and shaking with a small quantity of chloroform, the chloroform should not be colored violet. (Indicating less than 0.0005 per cent as I.)

Earths. — Dilute 10 c.c. of nitric acid with 80 c.c. of water and render slightly alkaline with ammonia water. On adding ammonium oxalate solution no turbidity should result. (Indicating less than 0.0002 per cent earths as Ca.)

Arsenic. — 50 c.c. of nitric acid are mixed with 5 c.c. of arsenic-free sulphuric acid (sp.gr. 1.84) and evaporated as far as possible on a water-bath, and then heated on a sand-bath until fumes of sulphur trioxide are given off. A Marsh apparatus is started, using 20 gm. of arsenic-free, granulated zinc and dilute (1 : 5) sulphuric acid; then the residue from the above evaporation, diluted with 20 c.c. of water, is washed into the generating flask of the apparatus. After the apparatus has been in action for one hour no mirror of arsenic should be visible. (Indicating less than 0.0000075 per cent As.)

Quantitative Determination. — Dilute 2 gm. of nitric acid with 50 c.c. of water and titrate with normal potassium hydroxide solution, using methyl orange as indicator.

1 c.c. of normal KOH = 0.06302 gm. of HNO_3, log. 79948.

II

ACID NITRIC, Sp.Gr. 1.30

A clear, colorless liquid, of specific gravity 1.30, and containing about 47 per cent of HNO_3.

TESTS OF PURITY

The tests to be made are those given under Acid Nitric, Sp.Gr. 1.40–1.42, observing the conditions there described. But instead of 1 c.c. of the acid, sp.gr. 1.40–1.42, use 1.5 c.c. of the acid sp.gr. 1.30.

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III

ACID NITRIC, Sp.Gr. 1.20

A clear, colorless liquid of specific gravity 1.20, and containing about 33 per cent of HNO_3.

TESTS OF PURITY

The tests to be made those given under Acid Nitric, Sp.Gr. 1.40–1.42, observing the conditions there described. But, instead of 1 c.c. of acid, sp.gr. 1.40–1.42, use 2 c.c. of acid sp.gr. 1.20.

IV

ACID NITRIC, Sp.Gr. 1.150–1.152

A clear, colorless liquid, of specific gravity 1.150–1.152 containing about 25 per cent of HNO_3.

TESTS OF PURITY

The tests to be made are those given under Acid Nitric, Sp.Gr. 1.40–1.42, observing the conditions there described. But, instead of 1 c.c. of acid, sp.gr. 1.40–1.42, use 2.5 c.c. of acid sp.gr. 1.150–1.152.

ACID NITRIC, CRUDE, Sp.Gr. 1.38–1.40

A clear, colorless, or yellowish liquid of specific gravity 1.38–1.40, and containing 61 to 65 per cent of HNO_3.

TEST OF PURITY

Non-volatile Matter. — 5 c.c. of crude nitric acid on evaporation should leave no weighable residue. (Indicating less than 0.007 per cent.)

ACID NITRIC, FUMING, Sp.Gr. 1.486–1.500*

A colorless or slightly yellowish liquid of specific gravity 1.486–1.500, and containing 87.7 to 94 per cent of nitric acid, HNO_3. Exposed to light it soon becomes brown, and then gives off suffocating yellowish-red vapors.

TESTS OF PURITY

The tests of purity to be made are those given under Acid Nitric, Sp.Gr. 1.40–1.42. But for 5 c.c. of acid sp.gr. 1.40–1.42 use 3 c.c. of fuming nitric acid.

* See foot-note under Acid Nitric, Sp.Gr. 1.40–1.42.

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ACID OXALIC, CRYSTALS

$H_2C_2O_4+2H_2O$. Mol. Wt. 126.05

Colorless, odorless, prismatic crystals, soluble in 10 parts of cold water, in about 3 parts of boiling water, and in 2.5 parts of alcohol; difficultly soluble in ether. On heating the hydrated oxalic acid in a capillary tube, it liquefies at 98° C. in its water of crystallization. On being heated to 70° C., the hydrated acid is rendered anhydrous, sublimes at about 100° C., and melts at 187° C. The crystals must contain 99.8–100 per cent of the hydrated acid, $H_2C_2O_4+2H_2O$, and must not appear effloresced.

TESTS OF PURITY

Ash. — 3 gm. of oxalic acid upon ignition in a platinum crucible should leave no weighable residue. (Indicating less than 0.017 per cent.)

Sulphuric Acid. — Dissolve 5 gm. of oxalic acid in 100 c.c. of water, and add 1 c.c. of hydrochloric acid and 1 c.c. barium chloride solution. No precipitate of barium sulphate should form on standing fifteen hours. (Indicating less than 0.004 per cent SO_3.)

Chlorides. — Dissolve 5 gm. of oxalic acid in 50 c.c. of water, add 15 c.c. of nitric acid and a few drops of silver nitrate solution. The solution may exhibit at most a slight opalescent turbidity. (Indicating less than 0.0004 per cent Cl.)

Heavy Metals. — The 1 : 10 aqueous solution must be perfectly clear, and on adding hydrogen sulphide water to 30 c.c. of it, no change should be observed. On now adding ammonia water to this liquid until alkaline, neither a green nor a brown coloration should develop, nor should a precipitate form. (Indicating none present.)

Ammonium Compounds. — On dissolving 2.5 gm. of oxalic acid and 5 gm. of potassium hydroxide (purest) in 30 c.c. of water, and adding to the solution about 15 drops of Nessler's solution, at most a slight yellow color should develop, but never a brownish-red one. (Indicating less than 0.002 per cent NH_3.)

Nitric Acid. — On overlaying 10 c.c. of a solution of diphenylamine* in concentrated sulphuric acid with 10 c.c. of a 1 : 10 aqueous solution of oxalic acid, a blue zone should not form at the contact-surfaces of the two liquids. (Indicating less than 0.007 per cent N_2O_5.)

Quantitative Determinations. — (*a*) Acidimetrically: Dissolve 1 gm. of crystallized oxalic acid in water, dilute to 100 c.c., and titrate 25 c.c. of the solution with one-fifth normal potassium hydroxide solution, using phenolphthalein as indicator. Perform the titration at 60° C.

1 c.c. of one-fifth normal KOH = 0.012605 gm. of $H_2C_2O_4+2H_2O$, log. 10054.

(*b*) By Oxidimetry: To 25 c.c. of the aqueous solution (1 gm. of crystallized oxalic acid in water as above) add 6 to 8 c.c. of concentrated sulphuric acid, heat to about 60° C., and titrate with decinormal potassium permanganate solution.

1 c.c. of decinormal $KMnO_4$ = 0.0063025 gm. of $H_2C_2O_4+2H_2O$, log. 79950.

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* See footnote under Diphenylamine.

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ACID PERCHLORIC

$HClO_4$. Mol. Wt. 100.47

A colorless liquid of specific gravity 1.12, and containing about 20 per cent of $HClO_4$.

TESTS OF PURITY

Non-volatile Matter. — 10 gm. of perchloric acid when evaporated and ignited should leave no weighable residue. (Indicating less than 0.005 per cent.)

Sulphuric Acid. — On diluting 5 c.c. of perchloric acid with 100 c.c. of water, and adding 5 c.c. of hydrochloric acid, followed by barium chloride solution, a precipitate of barium sulphate should not form on standing fifteen hours. (Indicating less than 0.005 per cent SO_3.)

Hydrochloric Acid. — The mixture obtained by diluting 5 c.c. of perchloric acid with 25 c.c. of water and adding 3 c.c. of nitric acid should not be rendered more than slightly turbid by silver nitrate solution. (Indicating less than 0.0005 per cent Cl.)

Barium. — 10 c.c. of perchloric acid diluted with 50 c.c. of water must not become turbid within five minutes on adding dilute sulphuric acid. (Indicating less than 0.0025 per cent Ba.)

Heavy Metals. — 10 c.c. of perchloric acid diluted with 40 c.c. of water must show no change on adding hydrogen sulphide water; and on adding to this solution 10 c.c. of ammonia water and a few drops of ammonium sulphide solution, a precipitate should not form, nor should the liquid acquire a brown color. (Indicating none present.)

ACID PHOSPHOMOLYBDIC

$20MoO_3 \cdot 2H_3PO_4 + 48H_2O$. Mol. Wt. 3940.89

Yellow, lustrous crystals, easily and completely soluble in water, and yielding with the latter an acid solution.

TESTS OF PURITY

Solubility, Heavy Metals, and Earths. — 1 gm. of phosphomolybdic acid should completely dissolve in 10 c.c. of water upon gentle warming. On adding to this solution two or three drops of ammonia water, a yellow precipitate forms, which completely redissolves on the addition of 5 c.c. of the ammonia water. On now adding to this solution ammonium sulphide solution, a precipitate should not form. At most a slight green color may appear. (Indicating at most a trace of heavy metals.) The addition of ammonium oxalate solution to this latter mixture should cause no visible change. (Indicating less than 0.02 per cent earths as Ca.)

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ACID PHOSPHORIC, ORTHO-

H_3PO_4. Mol. Wt. 98.06

I

ACID PHOSPHORIC, Sp.Gr. 1.70.

A clear, colorless, odorless, syrupy liquid, of specific gravity 1.70, and containing about 83 per cent of H_3PO_4.

TESTS OF PURITY

Volatile Acids. — Mix 30 c.c. of the phosphoric acid with 50 c.c. of water in a distilling flask. Distil* off 50 c.c., and titrate the distillate with decinormal potassium hydroxide solution, using methyl orange as indicator. Not more than 0.1 c.c. of the decinormal alkali solution should be required. (Indicating less than 0.00125 per cent as HNO_3.)

Nitric Acid. — 2 c.c. of phosphoric acid mixed with 2 c.c. of concentrated sulphuric acid, and overlaid with 1 c.c. of a solution of ferrous sulphate, must not develop a colored zone. (Indicating less than 0.0048 per cent N_2O_5.)

Hydrogen-Halogen Acids and Phosphorous Acid. — 2 c.c. of phosphoric acid diluted with 18 c.c. of water should give no reaction with silver nitrate solution either in the cold or on boiling. (Indicating less than 0.0003 per cent hydrogen-halogen acids as Cl, and less than 0.095 per cent P_2O_3.)

Sulphuric Acid. — On adding barium chloride solution to 20 c.c. of the diluted acid (1 : 10), no precipitate of barium sulphate should form on standing two or three hours. (Indicating less than 0.0025 per cent SO_3.)

Metaphosphoric Acid. — On dropping the acid diluted with 10 volumes of water into a dilute solution of albumen, no turbidity should ensue. (Indicating none present.)

Heavy Metals, Earths, etc. — (*a*) Dilute the acid with 10 volumes of water, and to 20 c.c. of the mixture add hydrogen sulphide water. There should be no visible change. (Indicating no heavy metals present and less than 0.01 per cent earths as Ca.)

(*b*) On adding 10 c.c. of ammonia water to 20 c.c. of the above diluted acid, no precipitate should form on the further addition of either ammonium oxalate solution (indicating less than 0.006 per cent earths as Ca), or ammonium sulphide solution. (Indicating no heavy metals present.)

(*c*) A mixture of 5 c.c. of phosphoric acid with 20 c.c. of absolute alcohol should remain perfectly clear. (Indicating no earths, etc. present.)

Substances Oxidizable by Permanganate. — On adding 5 drops of decinormal potassium permanganate solution to a mixture of 5 c.c. of phosphoric acid and 5 c.c. of diluted sulphuric acid, and then heating for 5 minutes at 100° C., the red color of the liquid should not disappear. (Indicating none present.)

Arsenic. — A Marsh apparatus is started, using 20 gm. of arsenic-free, granulated zinc, and diluted (1 : 5) sulphuric acid. A mixture of 3 c.c. of phosphoric acid with 20 c.c. of water is then introduced in small quantities at a time, the evolution of hydrogen being maintained for one hour. A deposit of arsenic should not be visible in the reduction tube within this time. (Indicating less than 0.0002 per cent As.)

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* In order to prevent spirting over of phosphoric acid, the flask should be provided with a Kjeldahl connecting bulb tube. The distillate must be tested with ammonium molybdate solution for phosphoric acid.

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Quantitative Determination. — Dilute 1 gm. of phosphoric acid with 30 c.c. of water, and titrate with normal potassium hydroxide, using phenolphthalein as indicator.* At least 17 c.c. of normal alkali must be added to produce the red color.

1 c.c. of normal KOH = 0.049032 gm. of H_3PO_4, log. 69048.

II

ACID PHOSPHORIC, Sp.Gr. 1.12

A clear, colorless, odorless liquid of specific gravity 1.12, and containing about 20 per cent of H_3PO_4.

TESTS OF PURITY

The tests to be made and the conditions to be observed are those given under Acid Phosphoric, Sp.Gr. 1.70. But for 1 c.c. of the phosphoric acid sp.gr. 1.70, use 4 c.c. of the phosphoric acid sp.gr. 1.12.

(ACID) PHOSPHORIC ANHYDRIDE

(Phosphorus Pentoxide)

P_2O_5. Mol. Wt. 142.08

A white, amorphous, odorless, bulky powder, which dissolves in water with a hissing noise, forming metaphosphoric acid. Phosphoric anhydride completely sublimes on being heated.

TEST OF PURITY

Arsenous Acid. — Introduce 1 gm. of phosphoric anhydride, in small portions at a time, into 20 c.c. of water, and while warming the solution, pass into it a current of hydrogen sulphide gas. The liquid may acquire at most a slightly yellow color, but a yellow precipitate should not form. (Indicating less than 0.01 per cent As_2O_3.)

ACID PHOSPHOTUNGSTIC

$(P_2O_5 \cdot 20WO_3 \cdot 11H_2O) + 16H_2O$. Mol. Wt. 5268.51

Small, white, or slightly yellowish-green crystals, easily soluble in water, and free from ammonia and nitric acid.

TESTS OF PURITY

Nitrates. — Dissolve 1 gm. of phosphotungstic acid in 10 c.c. of water, add a granule of sodium chloride and 1 drop of indigo solution (1 : 1000), followed

* Methyl orange may be used instead of phenolphthalein. In this case, however, 1 c.c. of the normal KOH = 0.09806 gm. of H_3PO_4, log. 99149. When titrating with phenolphthalein, twice as many cubic centimeters of the alkali solution are required as when using methyl orange.

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by 10 c.c. of concentrated sulphuric acid. The blue color of the mixture must not disappear within 10 minutes. (Indicating less than 0.0032 per cent N_2O_5.)

Ammonium Salts. — On heating a solution of 1 gm. of phosphotungstic acid in 10 c.c. of water, with 5 c.c. of sodium hydroxide solution (sp.gr. 1.3), no ammonia should be evolved (to be ascertained by means of moistened litmus paper). (Indicating less than 0.0045 per cent NH_3.)

NOTE.—Regarding a method for the quantitative analysis of phosphotungstic acid, see F. Kehrmann, Ber., **20**, 1813 (1887); *abst.*, J. Chem. Soc., **52**, 777 (1887).

ACID PICRIC

(Picronitric Acid; Trinitrophenol)

$C_6H_2(OH)(NO_2)_3$. Mol. Wt. 229.05

Pale yellow, glistening crystals, melting at 122.5° C., soluble in 100 parts of cold water and in about 30 parts of boiling water. The acid is soluble in 15 parts of alcohol, in 75 parts of ether, and in 20 parts of benzene.

TESTS OF PURITY

Resins: Substances Insoluble in Water. — 1 gm. of picric acid should dissolve completely and without turbidity in 100 c.c. of water. On adding to the solution 1 or 2 drops of 16 per cent sulphuric acid, no precipitate should form on standing fifteen hours. On subsequently filtering the liquid, no resin should remain on the filter. (Indicating none present.)

Picrates of Potassium, Sodium, and Ammonium.* — 1 gm. of picric acid must completely dissolve in 20 c.c. of benzene, affording a clear solution. (Indicating none present.)

Oxalic Acid. — On adding calcium chloride solution to a solution of 1 gm. of picric acid in 100 c.c. of water, a precipitate of calcium oxalate should not form within two hours. (Indicating less than 0.1 per cent $H_2C_2O_4$.)

Free and Combined Sulphuric Acid. — Evaporate a mixture of 2 gm. of picric acid and 10 c.c. of nitric acid (sp.gr. 1.4) to dryness on the water-bath. Dissolve the residue in 100 c.c. of boiling water with the addition of 5 c.c. of nitric acid, allow to cool, then filter, and add barium nitrate solution to the filtrate. There should be no immediate turbidity. (Indicating less than 0.25 per cent SO_3.)

Inorganic Matter (Ash). — 1 gm. of picric acid cautiously incinerated in an open platinum dish should not leave a residue weighing more than 0.001 gm. (Indicating less than 0.1 per cent.)

Quantitative Determination. — Titrate 1 gm. of picric acid dissolved in 100 c.c. of warm water with fifth normal potassium hydroxide solution, using phenolphthalein as indicator.

1 c.c. fifth normal KOH = 0.04581 gm. $C_6H_2(OH)(NO_2)_3$, log. 66096.

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* Picric acid cannot be tested for ammonia by warming with sodium hydroxide solution, as under such conditions picric acid itself decomposes with the evolution of ammonia.

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ACID ROSOLIC

(Corallin)

$C_{20}H_{16}O_3$. Mol. Wt. 304.12

Brittle, amorphous, reddish-brown pieces, presenting a metallic luster, and readily soluble in alcohol, but almost insoluble in water.

Rosolic acid is used as an indicator in the form of a solution of 0.5 gm. of the acid in a mixture of 50 c.c. of alcohol (about 85 per cent by weight) and 50 c.c. of water.

TEST OF SENSITIVENESS

Add 2 or 3 drops of the above rosolic acid solution to 100 c.c. of distilled water. On adding to the solution 0.05 c.c. of decinormal potassium hydroxide, the pale-yellow color of the water should change to a rose-red, and on the further addition of 0.05 c.c. of decinormal hydrochloric acid, the original color should be restored.

ACID SUCCINIC

$C_4H_6O_4$. Mol. Wt. 118.05

Colorless, monoclinic prisms, soluble in 20 parts of cold and about 2 parts of boiling water; in 10 parts of alcohol and in 80 parts of ether. Succinic acid melts at 182° C., and at 235° C. it boils with the evolution of white acrid fumes, while it undergoes decomposition to a large extent into water and succinic anhydride.

TESTS OF PURITY

Non-volatile Matter. — 1 gm. of succinic acid carefully heated in a platinum dish should volatilize and leave no weighable residue; nor should any charring occur. (Indicating less than 0.05 per cent.) The acid should not be allowed to burn.

Oxalic Acid. — The solution of 1 gm. of succinic acid in 20 c.c. of water should not be affected by calcium chloride solution. (Indicating less than 0.07 per cent $H_2C_2O_4$.)

Tartaric Acid.—Dissolve 5 gm. of succinic acid in 50 c.c. of warm water, add 5 c.c. of potassium acetate solution, and then mix the solution with 50 c.c. of alcohol. No precipitate should form on standing fifteen hours. (Indicating less than 2 per cent $H_2C_4H_4O_6$.)

Sulphates. — On adding barium nitrate solution to a solution of 1 gm. of succinic acid in 20 c.c. of water, no precipitate should form on standing fifteen hours. (Indicating less than 0.001 per cent SO_3.)

Chlorides. — On adding 2 or 3 c.c. of nitric acid to 20 c.c. of the 1 : 20 aqueous solution of succinic acid, not more than a faint opalescent turbidity should develop on the addition of silver nitrate solution. (Indicating less than 0.002 per cent Cl.)

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Ammonium Salts. — 1 gm. of succinic acid, on being heated with 10 c.c. of sodium hydroxide solution (sp.gr. 1.3), should liberate no ammonia (to be ascertained with moist litmus paper). (Indicating less than 0.0035 per cent NH_3.)

Heavy Metals. — 1 gm. of succinic acid dissolved in 20 c.c. of water should not be visibly changed by hydrogen sulphide water. (Indicating none present.)

Quantitative Determination. — Dissolve 1 gm. of succinic acid in 50 c.c. of water, and titrate with normal potassium hydroxide, using phenolphthalein as indicator.

1 c.c. of normal KOH = 0.059025 gm. of $C_4H_6O_4$, log. 77103.

ACID SULPHANILIC

$C_6H_4(NH_2)(SO_3H)1:4+2H_2O$. **Mol. Wt. 209.17**

Sulphanilic acid occurs in colorless, acicular, efflorescent crystals. It has no melting-point, but carbonizes on being heated to 280° to 300° C. The acid is difficultly soluble in cold water (about 160 parts), but more readily soluble in hot water; it is almost insoluble in alcohol, ether, and benzene.

TESTS OF PURITY

Inorganic Matter. — 1 gm. of sulphanilic acid should leave no weighable residue on ignition. (Indicating less than 0.05 per cent.)

Sulphuric Acid (Aniline Sulphate). — A solution of 1 gm. of sulphanilic acid acid in 25 c.c. of boiling water should not change in appearance on adding a few drops of barium chloride solution. (Indicating less than 0.004 per cent SO_3.)

Hydrochloric Acid (Aniline Hydrochloride). — On shaking 1 gm. of sulphanilic acid with 20 c.c. of water and filtering, the filtrate should exhibit at most a faint opalescent turbidity on the addition of a few drops of nitric acid and silver nitrate solution. (Indicating less than 0.002 per cent Cl.)

ACID SULPHURIC

H_2SO_4. **Mol. Wt. 98.09**

I

ACID SULPHURIC, Sp.Gr. 1.84

(Concentrated Sulphuric Acid)

A clear, colorless, oily liquid of specific gravity 1.84, and containing 95 to 96 per cent of H_2SO_4.

TESTS OF PURITY

Non-volatile Matter. — 100 gm. of sulphuric acid, on being evaporated and ignited, should leave no weighable residue. (Indicating less than 0.0005 per cent.)

Nitric Acid. — Dilute 5 c.c. of sulphuric acid with 5 c.c. of water, and overlay this liquid on 5 c.c. of diphenylamine solution (see Diphenylamine, page 79). A blue zone should not form at the contact-surfaces of the two liquids within one hour. (Indicating less than 0.0008 per cent N_2O_5.)

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Selenium. — On overlaying 2 c.c. of sulphuric acid with 2 c.c. of hydrochloric acid in which a granule of sodium sulphite has been dissolved, a reddish zone must not form at the contact-surfaces of the two liquids; nor should a red precipitate form on warming. (Indicating less than 0.0033 per cent Se.)

Substances Oxidizable by Permanganate (Nitrous and Sulphurous Acids). — Dilute 20 c.c. of sulphuric acid with 60 c.c. of water, and color the solution by adding 1 drop of decinormal potassium permanganate solution. The pink color should not disappear within five minutes. (Indicating less than 0.001 per cent as SO_2.)

Hydrogen-Halogen Acids. — 20 c.c. of sulphuric acid diluted with 20 c.c. of water should not become turbid within three hours on adding silver nitrate solution. (Indicating less than 0.0003 per cent as Cl.)

Lead. — On cautiously diluting 10 c.c. of sulphuric acid with 50 c.c. of alcohol (about 85 per cent by weight), no turbidity should be observed; nor should a precipitate of lead sulphate form on standing two hours. (Indicating less than 0.003 per cent Pb.)

Heavy Metals and Calcium. — (*a*) Dilute 10 c.c. of sulphuric acid with 50 c.c. of water, and add ammonia water in excess. On now adding a few drops of ammonium sulphide and ammonium oxalate solutions, neither a green color nor a turbidity should appear. (Indicating no heavy metals present, and less than 0.0055 per cent Ca.)

(*b*) On diluting 20 c.c. of sulphuric acid with 100 c.c. of water, and passing a current of hydrogen sulphide gas into the solution, no brown color should develop, nor should a precipitate form. (Indicating no heavy metals present.)

Ammonium Salts. — Dilute 2 c.c. of sulphuric acid with 30 c.c. of water, and add aqueous solution of potassium hydroxide (1 : 6) to alkalinity, followed by 10 to 15 drops of Nessler's reagent. At most a light-yellow color or precipitate may develop, but in no case should the color or precipitate be brownish-red. (Indicating less than 0.0015 NH_3.)

Arsenic. — Evaporate 200 gm. of the acid after adding a few cubic centimeters of nitric acid (1.40–1.42), to about 10 c.c., and dilute with 40 c.c. of water. A Marsh apparatus is set in operation, using 20 gm. of arsenic-free, granulated zinc, and arsenic-free, dilute sulphuric acid (1 : 5). As soon as the air has been expelled from the apparatus, heat the reduction tube to redness, and then gradually introduce the cooled acid mixture to be tested. No deposit of arsenic should be visible in the reduction tube within two hours. (Indicating less than 0.0000025 per cent As.)

Quantitative Determination. — The content of H_2SO_4 is most readily ascertained by taking the specific gravity, and then referring to tables for percentage. Consult the tables of Lunge, Isler, and Naef, Chem.-Tech. Untersuch.-Meth., 5 ed., 1, 354 (1904); compare Watts' Dict. of Chem., 4, 621 (1894); also J. Soc. Chem. Ind., 24, 790 (1905) and Landolt-Börnstein-Roth, Physikalisch-Chemische Tabellen, 4 ed., 265 (1912). The acid content may also be ascertained by diluting 5 gm. with water to 50 c.c. and titrating 10 c.c. of this with normal potassium hydroxide solution, using methyl orange as indicator.

1 c.c. of normal KOH = 0.049043 gm. of H_2SO_4, log. 69057.

II

ACID SULPHURIC, DILUTED, 16 PER CENT

A colorless liquid of specific gravity 1.110 to 1.114, and containing about 16 per cent of H_2SO_4.

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TESTS OF PURITY

The tests to be made and conditions to be observed are those given under Acid Sulphuric, Sp.Gr. 1.84. But for 1 c.c. of the acid sp.gr. 1.84 use 6 c.c. of the acid sp.gr. 1.110.

(ACID) SULPHURIC ANHYDRIDE

(Sulphur Trioxide)

SO_3. Mol. Wt. 80.07

Long, transparent, colorless prisms, which melt at 15° C. to a clear oily liquid, boiling at 46° C. On long keeping at a temperature below 25° C., the sulphuric anhydride polymerizes to a modification which forms long, silky, felted needles, which melt above 50° C., and which at a higher temperature become converted into the trioxide in vapor form, solidifying at 15° C., and boiling at 46° C.

NOTE. — Regarding the quantitative determination of sulphuric anhydride, see the statements given under Acid Sulphuric, Fuming. Also R. Rosenlecher, "The Assay of Fuming Sulphuric Acid and of Sulphuric Anyhdride," Z. anal. Chem. **37**, 209 (1898); *abst.*, J. Chem. Soc., **74**, II, 404 (1898).

ACID SULPHURIC, FUMING

I

ACID SULPHURIC, FUMING. FREE FROM NITROGEN

A colorless, oily liquid, fuming in the air, and containing 8 to 10 per cent of free SO_3 (i.e., 83.1 to 83.5 per cent total SO_3.)

TESTS OF PURITY

Non-volatile Matter. — 3 c.c. of fuming sulphuric acid, on being evaporated and ignited, should leave no weighable residue. (Indicating less than 0.009 per cent.)

Nitric Acid. — Dilute 1 c.c. of fuming sulphuric acid with 9 c.c. of water, and overlay the fluid on 5 c.c. of diphenylamine solution (see Diphenylamine, p. 79). No blue zone should form at the contact-surfaces of the two liquids. (Indicating less than 0.004 per cent N_2O_5, equivalent to 0.001 per cent N.)

Ammonium Salts. — Carefully add 2 c.c. of fuming sulphuric acid, by drops, to 30 c.c. of water, and add aqueous solution* (1 : 6) of potassium hydroxide to alkalinity; on now adding 10 to 15 drops of Nessler's reagent, at most a pale yellow, but in no case may a brownish-red color or precipitate develop. (Indicating less than 0.002 per cent NH_3.)

Halogens. — Add 1 c.c. of fuming sulphuric acid, by drops, to 30 c.c. of water; on now adding a few drops of silver nitrate solution, the liquid must not

* Made from Potassium Hydroxide, Purest.

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acquire more than a faint opalescent turbidity. (Indicating less than 0.0015 per cent as Cl.)

Lead. — On cautiously adding 10 c.c. of fuming sulphuric acid, by drops, to 50 c.c. of alcohol (about 85 per cent by weight), a clear liquid should result, which should contain no precipitate of lead sulphate after standing two hours. (Indicating less than 0.003 per cent Pb.)

Arsenic. — A Marsh apparatus is set in operation, using 20 gm. of arsenic-free, granulated zinc, and arsenic-free, dilute (1 : 5) sulphuric acid. As soon as the air has been expelled from the apparatus, heat the reduction tube to redness, then run into the apparatus a cooled mixture of 10 c.c. of the fuming sulphuric acid with 90 c.c. of water. No deposit of arsenic should be visible in the reduction tube within an hour. (Indicating less than 0.0001 per cent As.)

Quantitative Determination.* — Weigh off accurately 1 gm. of fuming sulphuric acid in a Lunge-Rey stoppered bulb pipette,† and cautiously allow the acid to run into 10 c.c. of water, contained in a porcelain dish. The contents of the dish are now rinsed into a measuring flask of 100 c.c. capacity, and the flask then filled up with water to the mark. After vigorously shaking, 25 c.c. of the liquid are titrated with fifth normal potassium hydroxide solution, using methyl orange as indicator. ‡

1 c.c. of fifth normal KOH = 0.008007 gm. of SO_3, log. 90347.

II

ACID SULPHURIC, FUMING §

An oily liquid, sometimes slightly colored, and often not perfectly clear. The acid fumes in the air, contains from 8 to 10 per cent of free SO_3, and is intended for nitrogen determinations by the Kjeldahl method.

TESTS OF PURITY

Nitrogen. — Dilute 30 c.c. of the fuming sulphuric acid with 100 c.c. of water, add 3 gm. of zinc dust, and allow the mixture to stand with frequent stirring until the evolution of hydrogen has ceased. Then add nitrogen-free solution of sodium hydroxide (sp.gr. 1.3) until strongly alkaline, and distil, collecting the distillate in a receiver containing about 10 c.c. of water and 2 or 3 c.c. of fifth

* In accurate determinations, the acidity due to sulphurous acid must be deducted from that determined by titration. Regarding this, see Lunge, Chem.-tech., Untersuch.-Meth., 5 ed., 1, 395 (1904); compare J. Chem. Soc., 68, II, 413 (1895).

† See Lunge, Chem.-tech. Untersuch.-Meth., 5 ed., 1, 394 (1904); illustrated in trade catalogues of glassware, etc.

‡ In order to ascertain the free SO_3 from the total SO_3 found by analysis, see the table in Lunge, Chem.-tech. Untersuch.-Meth., 5 ed., 1, 399 (1904); compare Thorpe's Dic. of Appld. Chem., 2 ed., 3, 711 (1895).

§ This acid, which contains traces of nitric acid, is considerably cheaper than the foregoing acid, which is practically free from nitrogen. It is well adapted for use in laboratories where fuming sulphuric acid is very frequently used for the Kjeldahl nitrogen determinations. For accurate nitrogen determinations, however, it will be necessary to determine the nitrogen content of the acid by a blank test carried out in the manner above described.

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normal hydrochloric acid. The distillate is then titrated with fifth normal potassium hydroxide solution, using methyl orange as indicator. To neutralize the ammonia from the fuming sulphuric acid should not require more than 0.2 c.c. of the fifth normal hydrochloric acid. (Indicating not more than 0.001 per cent N.)

Quantitative Determination. — The determination is carried out as described under Acid Sulphuric, Fuming, Free from Nitrogen.

ACID SULPHURIC, WITH PHOSPHORIC ANHYDRIDE

Approximately 10, 15, or 20 per cent P_2O_5

Sulphuric acid containing phosphorus pentoxide. Approximately 10, 15, or 20 per cent of P_2O_5 are the usual strengths. The preparation is employed in Kjeldahl's nitrogen determination.

TESTS OF PURITY

Nitric Acid. — Dilute 1 c.c. of the solution of phosphoric anhydride in sulphuric acid with 9 c.c. of water, and overlay this fluid on 5 c.c. of diphenylamine solution (see Diphenylamine, page 79). No blue zone should form at the contact-surfaces of the two liquids. (Indicating less than 0.004 per cent N_2O_5.)

Ammonium Salts. — Dilute 2 c.c. of the solution of phosphoric anhydride in sulphuric acid with 30 c.c. of water, and add aqueous solution* of potassium hydroxide (1 : 6) to alkalinity, followed by 10 to 15 drops of Nessler's reagent. At most only a faint yellow, but in no case a brownish-red, color or precipitate should form. (Indicating less than 0.002 per cent NH_3.)

ACID SULPHURIC, FUMING, WITH PHOSPHORIC ANHYDRIDE

Approximately 5, 10, 15, 20, or 25 per cent P_2O_5

Fuming sulphuric acid containing phosphorus pentoxide; the usual grades contain approximately 5, 10, 15, 20, or 25 per cent of P_2O_5. The preparation is used in Kjeldahl's nitrogen determination.

TEST OF PURITY

Nitrogen. — The nitrogen determination is carried out as described under Acid Sulphuric, Fuming. See footnote there.

ACID SULPHUROUS

SO_2+Aq. Mol. Wt. 64.07

A clear, colorless liquid of specific gravity 1.029 to 1.035. The acid first reddens blue litmus paper, and then bleaches it. The liquid contains about 6 per cent of SO_2.

* Made from Potassium Hydroxide, Purest.

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TESTS OF PURITY

Non-volatile Matter. — 10 c.c. of sulphurous acid, on being evaporated and ignited, should leave no weighable residue. (Indicating less than 0.048 per cent.)

Quantitative Determination. — Weigh off 10 gm. of sulphurous acid in a measuring flask of 100 c.c. capacity, and fill with boiled water up to the mark. Allow the fluid to run from a burette into 30 c.c. of decinormal iodine solution, constantly shaken, until decoloration ensues. To effect this not more than 16.2 c.c. of the acid solution should be required.

1 c.c. of decinormal I = 0.003205 gm. of SO_2, log. 50562.

ACID, SULPHUROUS; CUBES FOR GENERATING

Cube-like pieces, containing at least 30 per cent of SO_2.

TEST OF STRENGTH

Quantitative Determination. — Introduce 1 gm. of the finely powdered cubes and 5 gm. of anhydrous sodium carbonate into a graduated flask of 100 c.c. capacity; add 50 c.c. of water, boil the mixture for about ten minutes, allow to cool, fill the flask up to the mark, mix, and filter. Run the filtrate from a burette into a constantly shaken mixture of 30 c.c. of decinormal iodine solution and 10 c.c. of hydrochloric acid, until complete decoloration ensues.

1 c.c. of decinormal I = 0.003205 gm. of SO_2, log. 50562.

ACID TANNIC

(Tannin)

$C_{14}H_{10}O_9 + 2H_2O$. Mol. Wt. 358.11

A yellowish powder, or crystal-like lustrous scales. Tannic acid is soluble in 1 part of water and in 2 parts of alcohol (about 85 per cent by weight), yielding a clear liquid acid to litmus paper. Tannic acid is also soluble in about 8 parts of glycerin, but is difficultly soluble in ether* (of sp.gr. 0.720). Solutions of tannic acid are dextrorotary.

TESTS OF PURITY

Inorganic Matter (Zinc). — 4 gm. of tannin on ignition should not leave a residue exceeding 0.005 gm. in weight. (Indicating less than 0.125 per cent inorganic matter.) If this residue is dissolved in 2 c.c. of acetic acid, then diluted with 8 c.c. of water and filtered, the filtrate should not show more than a slight opalescent turbidity when treated with hydrogen sulphide water. (Indicating less than 0.006 per cent Zn.)

Sugar, Dextrin. — On mixing 10 c.c. of an aqueous (1 : 5) solution of tannic acid with 10 c.c. of alcohol (about 85 per cent by weight) the mixture must

* The solubility in ether depends very largely upon the amount of alcohol in the ether.

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remain clear for one hour; and no turbidity should occur on the further addition of 5 c.c. of ether. (Indicating none present.)

Water. — On drying tannic acid at 100° C., it should not lose more than 12 per cent of its weight.

NOTE. — Regarding the quantitative determination of tannic acid see Fresenius, Anleitung zur quantitativen Analyse, 6 ed., **2**, 619, or Lunge, Chem.-tech. Untersuch.-Meth., 5 ed., **3**, 698 ff. (1905); Fresenius-Cohn, Quantitative Analysis, **2**, 767 ff. (1904). Compare Allen's Commercial Organic Analysis, 4 ed., **5**, 57 ff. (1911).

ACID TARTARIC

$C_4H_6O_6$. Mol. Wt. 150.05

Colorless, prismatic crystals, or crystalline crusts, soluble in 0.8 part of water, and in 4 parts of alcohol (about 85 per cent by weight).

TESTS OF PURITY

Sulphuric Acid and Calcium. — Separate 20 c.c. portions of the aqueous (1 : 10) solution of the acid should show no change with barium chloride or ammonium oxalate solution. (Indicating less than 0.005 per cent SO_3, and less than 0.01 per cent Ca.)

Oxalic Acid. — A solution of 2 gm. of the acid in 20 c.c. of water when brought to only slight acidity by adding ammonia water, should show no change on adding calcium sulphate solution. (Indicating less than 0.035 per cent $H_2C_2O_4$.)

Lead* and Other Metals. — (*a*) The solution of 5 gm. of tartaric acid in 20 c.c. of water with 12 c.c. of ammonia water added, should not develop a brown color on the addition of hydrogen sulphide water. (Indicating none present.)

(*b*) 20 c.c. of the 1 : 10 aqueous solution should not be affected by hydrogen sulphide water. (Indicating none present.)

Inorganic Matter. — 1 gm. of tartaric acid, on being ignited, should leave no weighable residue. (Indicating less than 0.05 per cent.)

Quantitative Determination. — Dissolve 1 gm. of tartaric acid in 50 c.c. of water, and titrate with normal sodium hydroxide solution, using phenolphthalein as indicator.

1 c.c. of normal NaOH = 0.075025 gm. of $C_4H_6O_6$, log. 87520.

ACID THIOACETIC

CH_3COSH. Mol. Wt. 76.10

A yellow liquid of penetrating odor, boiling between 92° and 97° C., and having a specific gravity of 1.070. Thioacetic acid is soluble in 16 parts of water, and easily in alcohol. The 6 per cent aqueous solution is used instead of hydrogen sulphide in chemical analysis.†

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* Regarding the examination of tartaric acid for lead see Tatlock and Thompson, Analyst, **33**, 173 (1908); *abst.*, C. A. **2**, 2269 (1908).

† Regarding the use of thioacetic acid, see the following: R. Schiff and N. P. Tarugi, Ber., **27**, 3437 (1894); *abst.*, J. Chem. Soc., **68**, II, 84 (1895). R. Schiff, Ber., **28**, 1204 (1895) [or Z. anal. Chem., **34**, 456 (1895)]; *abst.*, J. Chem. Soc., **68**, II, 370 (1895).

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TESTS OF PURITY

Non-volatile Matter. — 10 c.c. of thioacetic acid, on being evaporated and ignited, should leave no weighable residue. (Indicating less than 0.0047 per cent.)

Sulphuric Acid. — On dissolving 5 c.c. of thioacetic acid in 100 c.c. of water, and adding barium chloride solution, no turbidity should occur. (Indicating less than 0.003 per cent SO_3.)

ALCOHOL AMYLIC

$C_5H_{11}OH$. Mol. Wt. 88.10

I

AMYL ALCOHOL

(Iso-Amyl Alcohol)

A clear, colorless liquid, neutral to litmus paper. Amyl alcohol is but slightly soluble in water, but is clearly miscible with alcohol, ether, and benzene. It has a specific gravity of 0.814 and boils at 131° C.

TESTS OF PURITY

Non-volatile Matter. — 10 gm. of amyl alcohol evaporated on the water-bath should leave no weighable residue. (Indicating less than 0.005 per cent.)

Foreign Organic Matter (Furfural, etc.). — (*a*) On shaking 5 c.c. of amyl alcohol with 5 c.c. of concentrated sulphuric acid, the mixture should not acquire more than a faint yellow or reddish color. (Indicating none present.)

(*b*) On shaking 5 c.c. of amyl alcohol with 5 c.c. of potassium hydroxide solution (sp.gr. 1.3), the amyl alcohol should not acquire any color. (Indicating none present.)

II

AMYL ALCOHOL FOR GERBER'S FAT DETERMINATION

A colorless liquid boiling at 129° to 132° C., and of specific gravity about 0.815.

TEST OF PURITY

1 c.c. of amyl alcohol shaken with 10 c.c. of concentrated sulphuric acid, (sp.gr. 1.820 to 1.825) and 11 c.c. of water in a Gerber butyrometer, then centrifuged for two or three minutes, and then allowed to stand for twenty-four hours, must not exhibit an oily segregate.*

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* Compare N. Gerber and M. M. Craandijk, Milch Ztg., **27**, 611 (1898). Chem. Zentr., (5), **2**, II, 907 (1898).

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ALCOHOL ETHYLIC

C_2H_5OH. Mol. Wt. 46.05

I

ALCOHOL ABSOLUTE

A clear, colorless liquid of specific gravity not over 0.797, corresponding to at least 99.46 per cent by volume, or 99.11 per cent by weight, of anhydrous alcohol, C_2H_5OH. It boils at 78° C., and should not affect litmus paper.

TESTS OF PURITY

Residue. — 50 c.c. of alcohol, on being slowly evaporated, should leave no residue. (Indicating none present.)

Fusel Oil.* — On mixing 10 c.c. of alcohol and 30 c.c. of water in an Erlenmeyer flask, no turbidity or coloration should be observed, nor should any foreign odor be noticeable. (Indicating none present.)

A mixture of 10 c.c. of alcohol and 0.2 c.c. of 15 per cent potassium hydroxide solution, when evaporated down to 1 c.c., and supersaturated with dilute sulphuric acid, should not have the odor of fusel oil. (Indicating none present.)

On rubbing a few drops of alcohol between the hands, no unpleasant odor should be noticeable. (Indicating none present.)

Dilute 5 c.c. of alcohol with 5 c.c. of water and then add 25 to 30 drops of an alcoholic solution of salicylaldehyde (1 : 100), following this with the addition of 20 c.c. of concentrated sulphuric acid. The mixture after cooling must show no reddish or garnet-red color.† (Indicating none present.)

Molasses-alcohol. — On overlaying 5 c.c. of alcohol on 5 c.c. of concentrated sulphuric acid, no rose-red zone should form within one hour at the contact-surfaces of the two liquids. (Indicating none present.)

Aldehyde. — A mixture of 10 c.c. of alcohol with 10 c.c. of water and 2 c.c. of ammoniacal silver nitrate solution‡ protected from the light should show neither a turbidity nor a color within fifteen hours. (Indicating none present.)

Organic Impurities.§ — The red color of a mixture of 10 c.c. of alcohol and 1 drop of a 1 : 1000 potassium permanganate solution should not pass into yellow within ten minutes. (Indicating none present.)

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* Regarding the quantitative determination of fusel oil, see Lunge, Chem.-tech. Untersuch.-Meth., 5 ed., **3**, 571 ff. (1905); compare Allen's Commercial Organic Analysis, 4 ed. Vol. I, p. 188 ff, (1909).

† If the alcohol is free from fusel oil the mixture after cooling is lemon-yellow. The presence of the slightest trace of fusel oil gives the mixture a yellow color by transmitted light and a reddish color by reflected light.

‡ Made by mixing 10 c.c. of silver nitrate solution (1 : 20) with 5 c.c. of ammonia water.

§ A partial decoloration of potassium permanganate occurs even with purest alcohol. Compare also M. E. Barbet, J. Pharm. Chim. **19**, 413, 457 (1889) or Pharm. Ztg., **34**, 481 (1889). P. Cazeneuve, Chem. Ztg., **13**, Rep. 198 (1889); for similar article see J. Chem. Soc., **56**, 928 (1889). Lang, Chem. Ztg., **17**, 1544 (1893).

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Metals and Tannin. — On adding to 10 c.c. of alcohol 1 c.c. of ammonia water or 5 c.c. of hydrogen sulphide water, no coloration should develop. (Indicating none present.)

Acetone. — Shake for one minute a mixture of 6 c.c. of baryta water, 6 drops of mercuric chloride solution (1 : 20), and 2 c.c. of alcohol; then filter. The clear filtrate must not show a dark color upon the addition of solution of ammonium sulphide. (Indicating less than 0.02 per cent.)

Furfural. — A mixture of 10 c.c. of alcohol, 1 drop of aniline, and 5 drops of diluted acetic acid (about 30 per cent) should show no red color upon standing one hour. (Indicating less than 0.0001 per cent.)

NOTE. — On the estimation of methyl alcohol in the presence of ethyl alcohol see T. E. Thorpe and J. Holmes, J. Chem. Soc., 85, I, 1, (1904).

II

ALCOHOL, 95 PER CENT

A clear, colorless liquid having a sp.gr. of about 0.816 at 15.6° C., corresponding to about 95 per cent by volume of anydrous alcohol, C_2H_5OH. It should not affect the color of litmus paper.

TESTS OF PURITY

The tests of purity to be applied and the conditions to be observed are those given under Alcohol Absolute.

III

ALCOHOL, 90 PER CENT

This alcohol has a specific gravity of 0.830 to 0.834, and contains 87.35 to 85.80 per cent of alcohol by weight, or 91.29 to 90.09 per cent by volume of anhydrous alcohol, C_2H_5OH.

TESTS OF PURITY

The tests of purity to be applied and the conditions to be observed are those given under Alcohol Absolute.

ALCOHOL METHYLIC

CH_3OH. Mol. Wt. 32.03

A colorless, mobile liquid, readily miscible in all proportions with water, alcohol, and ether. Methyl alcohol has the specific gravity 0.798, and boils between 65° and 66° C. It should not redden blue litmus paper.

TESTS OF PURITY

Non-volatile Matter. — 30 c.c. of methyl alcohol evaporated on the water-bath should leave no weighable residue. (Indicating less than 0.002 per cent.)

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Acetone and Ethyl Alcohol. — Shake together thoroughly 50 c.c. of double normal sodium hydroxide solution and 5 c.c. of methyl alcohol in a mixing cylinder, and add, with repeated shaking, 25 c.c. of double normal iodine solution. No turbidity or flocculent precipitate should occur; nor should an odor of iodoform be perceptible even after warming the mixture at above 60° C. for half an hour. (Indicating less than 0.015 per cent acetone, and less than 1 per cent ethyl alcohol.)

Empyreumatic Substances. — 1 c.c. of methyl alcohol should dissolve without turbidity in 10 c.c. of water. (Indicating none present.)

On allowing 5 c.c. of concentrated sulphuric acid to drop into 5 c.c. of methyl alcohol while kept cold, the mixture should not acquire more than a slightly yellowish color. (Indicating none present.)

Aldehydes. — On shaking 10 c.c. of methyl alcohol with 10 c.c. of sodium hydroxide solution (sp.gr. 1.3), the mixture should remain colorless. (Indicating none present.)

Substances Oxidizable by Permanganate. — On adding 1 drop of decinormal potassium permanganate solution to 10 c.c. methyl alcohol, the red color of the liquid must not disappear within ten minutes. (Indicating none present.)

Chloroform. — No odor of isonitrile should be developed upon bringing to boiling a mixture of 10 c.c. of methyl alcohol with 2 or 3 drops of aniline and 10 c.c. of alcoholic solution of potassium hydroxide (1 : 10). (Indicating less than 0.01 per cent $CHCl_3$.)

NOTE. — Regarding the quantitative determination of methyl alcohol, as well as the quantitative determination of acetone in methyl alcohol, see Lunge, Chem.-tech. Untersuch.-Meth., 6 ed., **3**, 970, 972 (1911). Compare Allen's Commercial Organic Analysis, 4 ed., **1**, 91 (1909). Also G. Krämer, H. Strache, and J. Messinger as given in note under Acetone, p. 1.

ALPHANAPHTHOL

$C_{10}H_7OH$. Mol. Wt. 144.06

Colorless, lustrous needles, having a phenolic odor, and melting at 97° C. Alphanaphthol is difficultly soluble in cold water, more readily in warm water, and dissolves easily in alcohol, ether, benzene and chloroform.

TESTS OF PURITY

Organic Acids. — Shake 1 gm. of alphanaphthol with 100 c.c. of water and filter. The filtrate should not redden blue litmus paper. (Indicating none present.)

Organic Substances Insoluble in Sodium Hydroxide Solution. — 1 gm. of alphanaphthol should dissolve completely and clearly in a mixture of 5 c.c. of sodium hydroxide solution III, and 5 c.c. of water. (Indicating none present.)

Inorganic Matter. — No weighable residue should remain on igniting 1 gm. of alphanaphthol. (Indicating less than 0.05 per cent.)

ALPHANAPHTHYLAMINE

$C_{10}H_7NH_2$. Mol. Wt. 143.08

A white, crystalline powder, quickly becoming reddish in the air; almost insoluble in water, easily soluble in alcohol and in ether, and melting at 50° C.

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TESTS OF PURITY

Solubility. — 0.5 gm. of alphaaaphthylamine should dissolve in 25 c.c. of diluted acetic acid, forming a clear and colorless solution.

Non-volatile Matter. — On heating, 1 gm. of alphanaphthylamine should volatilize, leaving no weighable residue. (Indicating less than 0.05 per cent.)

ALUMINUM OXIDE

Al_2O_3. Mol. Wt. 102.2

A white, or nearly white, very bulky, hygroscopic powder,* used for Wislicenus'† determination of tannin and colors. Under a microscope magnifying about 30 to 60 diameters the powder appears in the form of partly transparent, flocculent masses, often fibrous-like, resembling vegetable tissue.

TESTS OF PURITY

Metallic Mercury and Aluminum. — On moistening the preparation with water or alcohol, it acquires a gray color if any aluminum or mercury is present; if pure, it remains perfectly white. No globules of mercury or particles of metallic aluminum should be visible under the microscope. (Indicating none present.)

Absorptive Power for Tannin and Colors. — 1 gm. of aluminum oxide freshly heated (by the method in the footnote) is vigorously shaken for five minutes with 200 c.c. of a solution of 4.5 gm. of pure hydrated tannin (corresponding to 3.9 gm. of water-free tannin) or about the same amount of purified congo red (free from chloride and sulphate of sodium) in sufficient water to make 1000 c.c. (Use a flask with a rubber stopper.) The mixture is at once heated for five minutes on the water-bath, and filtered through a paper 9 cm. in diameter. After rejecting the first 10 c.c. of the filtrate, 50 c.c. are evaporated in a weighed Philips' nickel dish‡ and the residue dried in a water-bath drying oven (Möslinger oven) four hours, after which it is allowed to stand an hour and a half in a closed dish in a desiccator, and is then weighed.

The total residue obtained in the same manner from 50 c.c. of the original tannin or dye solution, is also ascertained. On now calculating the residue for 200 c.c. of original solution, and deducting from this the total residue left from the solution treated with the aluminum oxide, the result should show that 1 gm. of aluminum oxide absorbs 0.40 to 0.50 gm. of tannin, or 0.035 to 0.045 gm. of congo red.

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* The preparation, just before use for quantitative absorption analysis must always be heated over a free flame until the bottom of the dish or crucible just begins to be red. Higher or longer heating reduces the absorptive power. If heated in rather thick layers the powder must take on a "boiling or seething" motion, and before quieting down must show the so-called crater formations.

† H. Wislicenus, Determination of Tannin without Hide Powder, Z. angew. Chem., **17**, 801 (1904); *abst.*, J. Soc. Chem. Ind., **23**, 765 (1904). Z. anal. Chem., **44**, 96 (1905); *abst.*, J. Chem. Soc., 88, II, 363 (1905). Z. Chem. Ind. Kolloide, **2**, Supplementheft, II, XI, (1908); *abst.*, J. Chem. Soc., **94**, II, 261 (1908). H. Wislicenus and W. Muth, Collegium, No. **225**, 157, No. **256**, 168; *abst.*, C. A., **1**, 2038 (1907).

‡ Pure nickel dish 8.5 cm. in diameter, with vertical sides 2.5 cm. high, and with a grooved, tightly-fitting cover.

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AMMONIA WATER

NH_3+H_2O. Mol. Wt. $NH_3 = 17.06$

The aqueous solution of ammonia gas is a clear, colorless liquid, the specific gravity of which decreases as the ammonia content increases.

For analytical purposes, two solutions of different concentration are ordinarily used, of which one has a specific gravity of about 0.96, and contains about 10 per cent of NH_3, while the other, the concentrated, has the specific gravity of about 0.925, and contains about 20 per cent of NH_3. In American laboratories a still more concentrated solution of ammonia is employed. Its strength is about 28 per cent and it is generally designated as "Stronger Ammonia Water."

I

AMMONIA WATER, 28 PER CENT

(Stronger Ammonia Water)

A clear, colorless liquid having a specific gravity of about 0.90 and containing about 28 per cent NH_3.

TESTS OF PURITY

Non-volatile Matter. — On evaporating 3.5 c.c. of the ammonia water on the water-bath, no weighable residue should remain. A trace of residue, however, is almost always left. (Indicating less than 0.016 per cent.)

Chlorides. — Dilute 3.5 c.c. of the ammonia water with 30 c.c. of water, and supersaturate with 20 c.c. of nitric acid. This liquid should show no change on the addition of silver nitrate solution. (Indicating less than 0.0003 per cent Cl.)

Pyridine. — Not more than a faint odor of pyridine should be perceptible on diluting 20 c.c. of ammonia water with 20 c.c. of water and nearly neutralizing with dilute sulphuric acid (1 : 4). (Indicating at most a trace present.)

Tar-bases (Aniline, Pyridine, Pyrrol, etc.). — Evaporate a mixture of 3.5 c.c. of the ammonia water and 20 c.c. of nitric acid on the water-bath. The residue on evaporation should have a pure white color. (Indicating none present.)

Heavy Metals. — On diluting 2 c.c. of the ammonia water with 20 c.c. of water, and then adding a few drops of ammonium sulphide solution, no change should appear. (Indicating none present.)

Sulphates. — Slightly acidify 3.5 c.c. of the ammonia water with hydrochloric acid, and add barium chloride solution. No precipitate of barium sulphate should form on standing fifteen hours. (Indicating less than 0.0025 per cent SO_3.)

Carbon Dioxide. — A mixture of 7 c.c. of the ammonia water with 20 c.c. of calcium hydroxide solution should not become more than very slightly turbid on boiling. (Indicating less than 0.015 per cent CO_2.)

Sulphides. — On adding a few drops of an ammoniacal lead acetate solution

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to 3.5 c.c. of the ammonia water, the liquid must not acquire a yellow or a brown color, nor should a dark precipitate form. (Indicating less than 0.001 per cent S.)

Calcium. — A mixture of 3.5 c.c. of the ammonia water with 20 c.c. of water should exhibit no turbidity on the addition of ammonium oxalate solution. (Indicating less than 0.002 per cent Ca.)

Magnesium. — On adding ammonium phosphate solution to 7 c.c. of the ammonia water, diluted to 20 c.c., no precipitate should form on standing two hours. (Indicating less than 0.001 per cent Mg.)

Phosphates. — To 7 c.c. of the ammonia water add 40 c.c. of nitric acid and 25 c.c. of ammonium molybdate solution. No yellow precipitate should form in the liquid on standing two hours at about 40° C. (Indicating less than 0.0015 per cent P_2O_5.)

Substances Oxidizable by Potassium Permanganate. — To a mixture of 7 c.c. of ammonia water and 40 c.c. of water, add 40 c.c. of diluted sulphuric acid and 0.1 c.c. of decinormal potassium permanganate solution. The pink color of the liquid should not disappear on boiling five minutes. (Indicating none present.)

Quantitative Determination. — Dilute 2 gm. of the ammonia water with about 50 c.c. of water, and titrate with normal solution of hydrochloric acid, using methyl orange as indicator.

1 c.c. normal HCl = 0.01703 gm. NH_3, log. 23121.

II

AMMONIA WATER, 20 PER CENT

(Concentrated Ammonia Water)

This solution of ammonia has the specific gravity 0.925, and contains about 20 per cent of NH_3.

TESTS OF PURITY

The tests of purity are the same as those given under Ammonia Water, 28 per cent. But instead of using 2 c.c., 3.5 c.c., and 7 c.c. of the 28 per cent, use 2.5 c.c., 5 c.c. and 10 c.c. respectively of the 20 per cent ammonia water.

III

AMMONIA WATER, 10 PER CENT

This is the article termed "Ammonia Water" throughout the text of this book. It contains about 10 per cent of NH_3, and has a specific gravity about 0.96.

TESTS OF PURITY

The tests of purity are the same as those given under Ammonia Water, 28 per cent. But instead of using 2 c.c., 3.5 c.c., and 7 c.c., of the 28 per cent, use 5 c.c., 10 c.c., and 20 c.c. respectively of the 10 per cent ammonia water.

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AMMONIUM ACETATE

$NH_4C_2H_3O_2$. Mol. Wt. 77.07

A white, hygroscopic, crystalline mass, easily soluble in water and in alcohol.

TESTS OF PURITY

Non-volatile Matter. — 3 gm. of ammonium acetate, when ignited, should leave no weighable residue. (Indicating less than 0.0167 per cent.)

Chlorides. — The solution of 1 gm. of ammonium acetate in 20 c.c. of water, acidulated with 5 c.c. of nitric acid, should show no change on the addition of silver nitrate solution. (Indicating less than 0.0005 per cent Cl.)

Sulphates. — On adding 1 c.c. of hydrochloric acid to a solution of 1 gm. of ammonium acetate in 20 c.c. of water, followed by barium chloride solution, no precipitate of barium sulphate should form on standing fifteen hours. (Indicating less than 0.0075 per cent SO_3.)

Heavy Metals and Earths. — The solution of 5 gm. of ammonium acetate in 100 c.c. of water should not be affected by hydrogen sulphide water. Furthermore, the addition of ammonia water and ammonium oxalate solution should cause neither a coloration nor a turbidity. (Indicating no heavy metals present, and less than 0.004 per cent earths as Ca.)

AMMONIUM CARBONATE

$(NH_4)HCO_3 \cdot (NH_4)NH_2CO_2$

The term "ammonium carbonate" is generally applied to a mixture of ammonium bicarbonate and carbamate, forming a crystalline, white, translucent mass which readily effloresces, becomes opaque, and gives off an odor of ammonia.

Ammonium carbonate is soluble in 5 parts of water, and contains at least 31.76 per cent of ammonia (NH_3).

TESTS OF PURITY

Non- volatile Matter. — 5 gm. of ammonium carbonate, when ignited, should leave no weighable residue. (Indicating less than 0.01 per cent.)

Calcium. — The solution of 2 gm. of ammonium carbonate in 20 c.c. of water and 10 c.c. of acetic acid (sp.gr. 1.040–1.042) should remain clear ten minutes after the addition of ammonium oxalate solution. (Indicating less than 0.0125 per cent Ca.)

Sulphates. — Dissolve 5 gm. of ammonium carbonate in 100 c.c. of water and 10 c.c. of hydrochloric acid, heat the solution to boiling, and add barium chloride solution. No precipitate of barium sulphate should form on standing fifteen hours. (Indicating less than 0.01 per cent SO_3.)

Chlorides and Thiosulphates. — The solution of 2 gm. of ammonium carbonate in 20 c.c. of water should not be affected on adding 10 c.c. of nitric acid, followed by silver nitrate solution. (Indicating less than 0.00025 per cent Cl, and less than 0.0008 per cent $[NH_4]_2S_2O_3$.)

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Phosphate. — A solution of 2 gm. of ammonium carbonate in 20 c.c. of water and 10 c.c. of nitric acid must show no yellow precipitate within fifteen minutes after the addition of 20 c.c. of ammonium molybdate solution, keeping the temperature about 40° C. (Indicating less than 0.01 per cent P_2O_5.)

Heavy Metals. — To a solution of 2 gm. of ammonium carbonate in 20 c.c. of water are added a few drops of ammonium sulphide solution. Neither a green nor a brown color should appear. (Indicating none present.)

No change should appear upon adding hydrogen sulphide water to a solution of 2 gm. of ammonium carbonate in 20 c.c. of water containing 10 c.c. of hydrochloric acid. (Indicating none present.)

Sulphocyanates. — The solution of 1 gm. of ammonium carbonate in 20 c.c. of water and 3 c.c. of hydrochloric acid should not be reddened on adding 1 drop of ferric chloride solution. (Indicating less than 0.12 per cent CNS.)

Tar Bases (Aniline, etc.). — Acidify 1 gm. of ammonium carbonate with 5 c.c. of nitric acid, and evaporate the solution to dryness on the water-bath. The residue must have a pure white color. (Indicating none present.)

Quantitative Determination. — Dissolve 2 gm. of ammonium carbonate (translucent undecomposed pieces) in 50 c.c. of water mixed with 50 c.c. of normal hydrochloric acid. Then titrate the excess of acid with normal potassium hydroxide solution, using methyl orange as indicator. At least 37.3 c.c. of the acid should be consumed by the carbonate.

1 c.c. of normal HCl = 0.01703 gm. of NH_3, log. 23121.

AMMONIUM CHLORIDE

NH_4Cl. Mol. Wt. 53.50

A white, crystalline powder, soluble in 3 parts of water, and in 60 parts of alcohol.

TESTS OF PURITY

Non-volatile Matter. — 5 gm. of ammonium chloride, when gently ignited, should leave no weighable residue. (Indicating less than 0.01 per cent.)

Phosphates and Arsenates. — Dissolve 5 gm. of ammonium chloride in 20 c.c. of water, and add to the clear solution 3 c.c. of magnesia mixture* and 10 c.c. of ammonia water. No precipitate should form on standing fifteen hours. (Indicating less than 0.001 per cent P_2O_5, and less than 0.005 per cent As_2O_5.)

Heavy Metals. — The solution of 2 gm. of ammonium chloride in 20 c.c. of water must remain clear after the addition of ammonia water, and must show neither a green nor a brown color upon the further addition of ammonium sulphide solution. (Indicating none present.)

The solution of 2 gm. of ammonium chloride in 20 c.c. of water must show no change upon the addition of a few drops of hydrochloric acid and some hydrogen sulphide water. (Indicating none present.)

Sulphates. — The solution of 5 gm. of ammonium chloride in 100 c.c. of water with 5 c.c. of hydrochloric acid and some barium chloride solution should show no precipitate of barium sulphate on standing fifteen hours. (Indicating less than 0.005 per cent SO_3.)

Sulphocyanates. — Dissolve 1 gm. of ammonium chloride in 20 c.c. of water, and add to the solution 2 c.c. of hydrochloric acid and 1 drop of ferric chloride solution. The liquid should not acquire a red color. (Indicating less than 0.12 per cent CNS.)

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* See note under Acid Hydrobromic (page 9) for formula.

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Tar Bases (Aniline, etc.) — On evaporating to dryness 1 gm. of ammonium chloride with 5 c.c. of nitric acid on the water-bath, the residue must have a pure white color. (Indicating none present.)

Calcium. — 2 gm. of ammonium chloride are dissolved in 20 c.c. of water. When 1 c.c. of ammonia water and some ammonium oxalate solution are added the mixture must remain clear ten minutes. (Indicating less than 0.01 per cent Ca.)

AMMONIUM CHROMATE

(Ammonium Chromate, Neutral)

$(NH_4)_2CrO_4$. Mol. Wt. 152.08

Yellow, needle-shaped crystals soluble in 5 parts of cold water.

TESTS OF PURITY

Alkalies. — Add 50 c.c. of solution of lead acetate (1 : 10 and free from alkalies) to a solution of 2 gm. of ammonium chromate in 100 c.c. of water. Filter off the precipitate of lead chromate, and remove the lead from the filtrate by passing hydrogen sulphide gas through it. Filter, and evaporate this (lead-free) filtrate to dryness. The residue thus obtained should not weigh more than 0.005 gm. after ignition. (Indicating not more than 0.25 per cent.)

Chlorides. — Add 10 c.c. of nitric acid to a solution of 1 gm. of ammonium chromate in 20 c.c. of water and warm to about 50° C. The addition of a few drops of solution of silver nitrate to this should produce neither a turbidity nor a precipitate within five minutes. (Indicating less than 0.0025 per cent Cl.)

Sulphates. — The addition of solution of barium chloride to a solution of 3 gm. of ammonium chromate in 100 c.c. of water and 30 c.c. of hydrochloric acid should cause no precipitate of barium sulphate within fifteen hours. (Indicating less than 0.029 per cent SO_3.)

Aluminum and Calcium. — The solution of 2 gm. of ammonium chromate in 30 c.c. of water upon the addition of 5 c.c. of ammonia water and some solution of ammonium oxalate should contain no precipitate at the end of fifteen hours. (Indicating less than 0.1 per cent Al and less than 0.005 per cent Ca.)

Quantitative Determination. — Dissolve 1 gm. of ammonium chromate in sufficient water to make 100 c.c. of solution, and of this place 10 c.c. in a glass-stoppered flask of about 400 c.c. capacity. Add 2 gm. of potassium iodide, 5 c.c. of diluted sulphuric acid (sp.gr. 1.110–1.114), and 350 c.c. of water. Titrate the liberated iodine with decinormal solution of sodium thiosulphate, using starch solution as indicator.

1 c.c. of decinormal solution of $Na_2S_2O_3 = 0.0050695$ gm. of $(NH_4)_2CrO_4$, log. 70497.

AMMONIUM CITRATE SOLUTION

A clear, colorless liquid, containing 150 gm. of pure, crystallized citric acid and 23 gm. of ammonia-nitrogen (= 27.96 gm. of NH_3) per liter. The solution has an acid reaction to litmus paper, and is used for determining citrate-soluble phosphoric acid in Thomas slag, by the method of Wagner.

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TEST FOR PROPER CONTENT OF AMMONIA-NITROGEN

Dilute 25 c.c. of the ammonium citrate solution with water to 250 c.c. To 25 c.c. of this diluted solution add 3 gm. of calcined magnesia and about 200 c.c. of water, and distil, using a receiver containing 40 c.c. of semi-normal sulphuric acid. After the distillation titrate the excess of acid with semi-normal potassium hydroxide solution, using methyl orange as indicator.

1 c.c. of semi-normal H_2SO_4 = 0.00705 gm. of N, log. 84541, = 0.008517 gm. of NH_3, log. 93029.

AMMONIUM DITHIOCARBONATE SOLUTION

$CO(SNH_4)_2$. Mol. Wt. 128.22

A yellow liquid of ammoniacal odor, and containing about 10 to 12 per cent of ammonium dithiocarbonate, about 8 per cent of ammonium chloride, and small quantities of ammonium sulphocyanate and ammonium sulphide. It is used as a substitute for hydrogen sulphide and ammonium sulphide.

TESTS OF PURITY

Non-volatile Matter. — 10 c.c. of ammonium dithiocarbonate solution, on being evaporated and ignited, should leave no weighable residue. (Indicating less than 0.005 per cent.)

Ammonium Carbonate. — On adding 3 c.c. of calcium chloride solution to 10 c.c. of the ammonium dithiocarbonate solution, no precipitate should form, even on warming. (Indicating less than 0.0045 per cent $[NH_4]_2CO_3$.)

NOTE. — Regarding the employment of ammonium dithiocarbonate see M. Vogtherr, Ber. d. pharm. Ges., 8, 232 (1898) [or Pharm. Zentralhalle, 39, 692 (1898)]; *abst.*, J. Chem. Soc., 78, II, 241 (1900).

AMMONIUM FLUORIDE

NH_4F. Mol. Wt. 37.04

White crystals, easily soluble in water, affording a solution which usually exhibits an acid reaction because of the presence of $NH_4F \cdot HF$.

TESTS OF PURITY

Non-volatile Matter. — 10 gm. of ammonium fluoride on ignition should leave no weighable residue. (Indicating less than 0.005 per cent.)

Chlorides. — On dissolving 2 gm. of ammonium fluoride in 20 c.c. of water in a platinum dish, and adding a few drops of nitric acid, followed by silver nitrate solution, the liquid must show at most a slight opalescence. (Indicating less than 0.001 per cent Cl.)

Sulphates and Silicofluorides. — Dissolve 5 gm. of ammonium fluoride in a platinum dish in 25 c.c. of water, and add 25 c.c. of hydrochloric acid, followed by 1 to 2 c.c. barium chloride solution. On now mixing this solution with 50 c.c. of alcohol, no turbidity should ensue. (Indicating less than 0.3 per cent SO_3, and less than 0.015% $(NH_4)_2SiF_6$.)

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Heavy Metals. — Dissolve 2 gm. of ammonium fluoride in 25 c.c. of water, in a platinum dish, add to the solution a few drops of hydrochloric acid and some hydrogen sulphide water. No change should appear. (Indicating none present.)

A solution of 2 g . of ammonium fluoride in 20 c.c. of water in a platinum dish is made alkaline with ammonia water. It should remain clear. The addition to this of a few drops of ammonium sulphide solution should produce at most a slight greenish coloration, but a precipitate should not form. (Indicating at most a trace.)

AMMONIUM MOLYBDATE

$(NH_4)_6Mo_7O_{24}+4H_2O$. Mol. Wt. 1236.32

Large, colorless, or slightly greenish crystals, very easily soluble in water. When heated they liberate ammonia and water, leaving molybdic anhydride.

TESTS OF PURITY

Phosphates. — 10 gm. of ammonium molybdate with 25 c.c. of water and 20 c.c. of ammonia water (sp.gr. 0.92) should afford a clear solution. Add this solution to 150 c.c. of nitric acid (sp.gr. 1.20)*. No yellow precipitate should form on standing fifteen hours at a temperature of about 40° C. (Indicating less than 0.0005 per cent P_2O_5.)

Heavy Metals. — Dissolve 2 gm. of ammonium molybdate in 5 c.c. of water and 5 c.c. of ammonia water, and to the solution add a few drops of ammonium sulphide solution. No green coloration should develop, nor should a precipitate form. (Indicating none present.)

Sulphates. — On dissolving 1 gm. of ammonium molybdate in 10 c.c. of water, and acidifying the solution with 5 c.c. of nitric acid,* no change should be observed on the further addition of barium nitrate solution. (Indicating less than 0.175 per cent SO_3.)

Chlorides. — 2 gm. of ammonium molybdate dissolved in 10 c.c. of water and acidulated with 10 c.c. of nitric acid* should show at most a slight opalescence upon the addition of silver nitrate solution. (Indicating less than 0.001 per cent Cl.)

Nitrate. — To a solution of 1 gm. of ammonium molybdate and a small crystal of sodium chloride in 10 c.c. of water are added 1 drop of a solution of indigo (1 : 1000) and 10 c.c. of sulphuric acid (sp.gr. 1.84). The blue color of the solution must not disappear upon mixing the liquids. (Indicating less than 0.0032 per cent N_2O_5.)

Quantitative Determination. — The content of molybdic anhydride may be approximately determined by gently igniting about 1 gm. of the salt until the ammoniacal odor has disappeared, and then weighing the residue of molybdic anhydride; it should amount to about 81 per cent. The accurate determination is carried out in the manner described under Acid Molybdic Anhydride, on page 14.

AMMONIUM MOLYBDATE SOLUTION

This solution, known as solution of acid molybdic according to Fresenius, is made by dissolving 150 gm. of ammonium molybdate

* The ammonium molybdate solution should be added to the nitric acid gradually and with constant rotation; never otherwise.

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in 1 liter of water, gently heating, and subsequently pouring the resulting liquid, with constant stirring, into 1 liter of nitric acid (sp.gr. 1.20).

AMMONIUM NITRATE

NH_4NO_3. Mol. Wt. 80.05

Colorless crystals very easily soluble in water.

TESTS OF PURITY

The tests to be made and conditions to be observed are those described under Ammonium Chloride; and, in addition, the following tests for chlorides and nitrites:

Chlorides. — On dissolving 2 gm. of ammonium nitrate in 20 c.c. of water, and adding a few drops of nitric acid, followed by silver nitrate solution, the liquid should remain unchanged. (Indicating less than 0.00025 per cent Cl.)

Nitrites. — To the solution of 1 gm. of ammonium nitrate in 20 c.c. of water are added 1 c.c. of 16 per cent sulphuric acid and 1 c.c. of a freshly prepared (1 : 100) colorless* solution of metaphenylenediamine hydrochloride; no yellow or yellowish-brown color should develop. (Indicating less than 0.00055 per cent N_2O_3.)

AMMONIUM OXALATE

$(NH_4)_2C_2O_4+H_2O$. Mol. Wt. 142.10

Colorless crystals, clearly soluble in 25 parts of cold water.

TESTS OF PURITY

Non-volatile Matter. — 3 gm. of ammonium oxalate on ignition should leave no weighable residue. (Indicating less than 0.017 per cent.)

Sulphates. — Dissolve 5 gm. of ammonium oxalate in 200 c.c. of water, heat the solution to boiling, and then add 10 c.c. of hydrochloric acid, followed by barium chloride solution. No precipitate of barium sulphate should form on standing fifteen hours. (Indicating less than 0.005 per cent SO_3.)

Chlorides. — On adding to a solution of 1 gm. of ammonium oxalate in 25 c.c. of water, 10 c.c. of nitric acid, and a few drops of silver nitrate solution, at most a slight opalescence should develop on shaking. (Indicating less than 0.0025 per cent Cl.)

Heavy Metals. — To a solution of 1 gm. of ammonium oxalate in 25 c.c. of water, add hydrogen sulphide water; the solution should remain unchanged. Now add to the liquid 5 c.c. of ammonia water; no green color should develop, nor should a precipitate form. (Indicating none present.)

Quantitative Determination. — Dissolve 1 gm. of ammonium oxalate in 100 c.c. of water. To 25 c.c. of this solution add 6 to 8 c.c. of sulphuric acid (sp.gr. 1.84), warm to about 60° C., and titrate with decinormal potassium permanganate solution.

1 c.c. of decinormal $KMnO_4$ = 0.007105 gm. of $(NH_4)_2C_2O_4+H_2O$, log. 85156.

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* Should the solution of metaphenylenediamine hydrochloride already have a color it is to be decolorized before use by warming with ignited animal charcoal.

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AMMONIUM PERSULPHATE

$(NH_4)_2S_2O_8$. Mol. Wt. 228.22

Colorless crystals, which dissolve very easily in water with the formation of a solution acid to litmus paper. The preparation contains at least 95 per cent of $(NH_4)_2S_2O_8$, although gradual deterioration occurs, oxygen being liberated.

TESTS OF PURITY

Non-volatile Matter. — Ignition of 3 gm. of ammonium persulphate should leave a residue not exceeding 0.002 gm. (Indicating not more than 0.0667 per cent.)

Chlorides. — The addition of 3 to 5 drops* of solution of silver nitrate to a solution of 1 gm. of ammonium persulphate in 20 c.c. of water should cause at most a slight opalescence. (Indicating less than 0.002 per cent Cl.)

Heavy Metals. — Evaporate to dryness on the water-bath a solution of 4 gm. of ammonium persulphate in 50 c.c. of sulphurous acid (sp.gr. 1.029–1.035), and dissolve the residue thus obtained in 40 c.c. of water.

Hydrogen sulphide water should produce no visible change in 20 c.c. of this solution. (Indicating none present.)

Ammonia water added in excess to the other 20 c.c. of the above solution of the residue from the evaporation should cause no visible change; and the subsequent addition of a few drops of solution of ammonium sulphide should produce at most a green color, in no case a precipitate. (Indicating at most a trace.)

Quantitative Determination. — In a graduated 100 c.c. flask place 1 gm. of ammonium persulphate and pour over it a solution of 5 gm. of potassium iodide in 50 c.c. of water, followed by 10 c.c. of diluted sulphuric acid (sp.gr. 1.110–1.114). Allow the mixture to stand with frequent shaking for about half an hour, then fill to the mark, mix, and titrate 20 c.c. of it with decinormal solution of sodium thiosulphate.

1 c.c. of decinormal $Na_2S_2O_3 = 0.011411$ gm. of $(NH_4)_2S_2O_8$, log. 05732.

AMMONIUM PHOSPHATE

(Secondary Ammonium Phosphate)

$(NH_4)_2HPO_4$. Mol. Wt. 132.13

Colorless crystals, or white, crystalline powder, soluble in 4 parts of cold, or 0.5 part of boiling, water. The aqueous solution is alkaline to litmus paper.

TESTS OF PURITY

Alkalies. — The solution of 2 gm. of ammonium phosphate in 100 c.c. of water is treated with lead acetate solution† in slight excess to precipitate the

* Larger quantities of the silver nitrate solution obscure the chloride reaction by a black precipitate.

† Must be free from alkalies, etc.

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phosphoric acid, and is then filtered. The excess of lead in the filtrate is then precipitated with hydrogen sulphide, the liquid filtered, the filtrate evaporated to dryness, and the residue ignited. The residue should not weigh more than 0.003 gm. (indicating not more than 0.15 per cent), nor should it have an alkaline reaction to litmus paper after moistening with a few drops of water.

Arsenic. — On shaking 1 gm. of powdered ammonium phosphate with 3 c.c. of stannous chloride solution, the mixture should not darken within one hour. (Indicating less than 0.0015 per cent As.)

Carbonates and Sulphates. — On adding hydrochloric acid to a solution of 1 gm. of ammonium phosphate in 20 c.c. of water, no effervescence should take place (indicating less than 2.0 per cent CO_2); and on adding barium chloride solution, no precipitate of barium sulphate should form on standing fifteen hours. (Indicating less than 0.0075 per cent SO_3.)

Chlorides. — On dissolving 1 gm. of ammonium phosphate in 20 c.c. of water, and adding 5 c.c. of nitric acid, followed by silver nitrate solution, not more than a very slight opalescence should develop. (Indicating less than 0.0015 per cent Cl.)

Nitrates. — Dissolve 2 gm. of ammonium phosphate and a small crystal of sodium chloride in 10 c.c. of water, and to the solution add 1 drop of indigo solution (1 : 1000), followed by 10 c.c. of concentrated sulphuric acid. The liquid should still retain its blue color upon shaking. (Indicating less than 0.0016 per cent N_2O_5.)

Heavy Metals. — On dissolving 2 gm. of ammonium phosphate in 20 c.c. of water, acidifying the solution with hydrochloric acid, and then adding hydrogen sulphide water, no change should be seen. Upon dissolving 2 gm. of ammonium phosphate in 20 c.c. of water and adding ammonium sulphide solution, no green color should develop, nor should a precipitate form. (Indicating none present.)

Quantitative Determination. — 1 gm. of ammonium phosphate is dissolved in 100 c.c. of water and titrated with normal hydrochloric acid, using methyl orange as indicator.

1 c.c. of normal HCl = 0.13213 gm. of $(NH_4)_2HPO_4$, log. 12100.

AMMONIUM SULPHATE

$(NH_4)_2SO_4$. **Mol. Wt. 132.15**

Colorless crystals, soluble in 2 parts of cold, and in 1 part of boiling, water; insoluble in alcohol.

TESTS OF PURITY

Non-volatile Matter. — 3 gm. of ammonium sulphate on ignition should leave no weighable residue. (Indicating less than 0.017 per cent.)

Chlorides. — The solution of 2 gm. of ammonium sulphate in 20 c.c. of water should show no change on acidifying with nitric acid and then adding silver nitrate solution. (Indicating less than 0.00025 per cent Cl.)

Heavy Metals. — The solution of 2 gm. of ammonium sulphate in 20 c.c. of water should remain unaffected after acidulating with hydrochloric acid and adding hydrogen sulphide water. (Indicating none present.)

The solution of 2 gm. of ammonium sulphate in 20 c.c. of water should remain clear upon the addition of ammonia water, and upon the further addition of ammonium sulphide solution neither a green nor a brown color should appear, nor should a precipitate form. (Indicating none present.)

Sulphocyanates. — Dissolve 1 gm. of ammonium sulphate in 20 c.c. of water; on adding to the solution 2 c.c. of hydrochloric acid and 1 drop of ferric chloride

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solution, the liquid should not acquire a red color. (Indicating less than 0.12 per cent CNS.)

Phosphates and Arsenates. — Dissolve 5 gm. of ammonium sulphate in 20 c.c. of water, and add to the clear solution 3 c.c. of magnesia mixture* and 10 c.c. of ammonia water. No precipitate should form on standing fifteen hours. (Indicating less than 0.001 per cent P_2O_5, and less than 0.005 per cent As_2O_5.)

Nitrates. — Dissolve 2 gm. of ammonium sulphate and a small crystal of sodium chloride in 10 c.c. of water, and add to the solution 1 drop of indigo solution (1 : 1000), followed by 10 c.c. of concentrated sulphuric acid. The liquid should retain its blue color upon shaking. (Indicating less than 0.0016 per cent N_2O_5.)

AMMONIUM SULPHIDE SOLUTION

(Ammonium Sulphydrate Solution)

A colorless or yellowish liquid, produced by passing hydrogen sulphide into ammonia water. It is strongly alkaline towards litmus paper.

TESTS OF PURITY

Arsenic, Antimony and Tin. — On adding hydrochloric acid to 50 c.c. of ammonium sulphide solution to acidity, hydrogen sulphide gas is liberated in copious amounts, but no colored precipitate should form. (Indicating less than 0.006 per cent As, 0.0007 per cent Sb, or 0.0013 per cent Sn.)

Non-volatile Matter. — On evaporating and igniting 10 c.c. of ammonium sulphide solution in a porcelain dish, no weighable residue should remain. (Indicating less than 0.005 per cent.)

Ammonium Carbonate. — On adding 3 c.c. of calcium chloride solution to 10 c.c. of ammonium sulphide solution, no precipitate should form, even on warming. (Indicating less than 0.005 per cent CO_2.)

Chloride. — To 20 c.c. of the filtrate obtained in the Quantitative Determination of Sulphidic Sulphur (see the following test) add nitric acid to acid reaction. No turbidity or precipitation should result. (Indicating less than 0.005 per cent Cl.)

Quantitative Determination of the Sulphidic Sulphur. — Dilute 10 gm. of ammonium sulphide solution to 100 c.c., place 5 c.c. of this dilution in a graduated flask of 100 c.c. capacity, add 20 c.c. of ammonia water and 50 c.c. of decinormal solution of silver nitrate, fill with water to the mark, mix the contents of the flask thoroughly, and filter. Mix 50 c.c. of the filtrate with 20 c.c. of nitric acid, and after adding 5 c.c. of cold saturated solution of ferric ammonium sulphate, titrate with decinormal solution of ammonium sulphocyanate.

1 c.c. decinormal $AgNO_3 = 0.0016035$ gm. S, log. 20506.

Quantitative Determination of the Ammonia. — Dilute 20 gm. of the ammonium sulphide solution to 500 c.c. To 50 c.c. of this dilution add 30 c.c. of Sodium Hydroxide Solution, Solution I, Free from Nitrogen, and distil about 50 c.c., collecting the distillate in a receiver containing 20 c.c. of normal hydrochloric acid solution. Titrate the distillate with normal solution of potassium hydroxide, using methyl orange as indicator.

1 c.c. normal $HCl = 0.01703$ gm. NH_3, log. 23121.

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* See note under Acid Hydrobromic (page 9) for formula.

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AMMONIUM SULPHOCYANATE

(Ammonium Thiocyanate)

NH_4SCN. Mol. Wt. 76.12

Colorless crystals, easily soluble in water and in alcohol.

TESTS OF PURITY

Non-volatile Matter. — 2 gm. of ammonium sulphocyanate, on being ignited, should leave no weighable residue. (Indicating less than 0.025 per cent.)

Substances Insoluble in Alcohol. — 1 gm. of ammonium sulphocyanate should completely dissolve in 10 c.c. of absolute alcohol, and yield a perfectly clear solution. (Indicating none present.)

Sulphates. — On adding a few drops of hydrochloric acid, followed by barium chloride solution, to a solution of 1 gm. of ammonium sulphocyanate in 20 c.c. of water, no reaction should be observed within five minutes. (Indicating less than 0.01 per cent SO_3.)

Heavy Metals. — On dissolving 1 gm. of ammonium sulphocyanate in 20 c.c. of water, and adding to the solution several drops of ammonium sulphide solution, no precipitate should form, nor should a brown or green color develop. (Indicating none present.)

Iron. — The solution of 1 gm. of ammonium sulphocyanate in 20 c.c. of water should remain perfectly colorless on adding a few drops of hydrochloric acid. (Indicating less than 0.0004 per cent Fe.)

AMMONIUM THIOACETATE SOLUTION

(Schiff's Reagent)

CH_3COSNH_4. Mol. Wt. 93.14

A clear, yellowish liquid, having a faint odor resembling that of ammonium sulphide, and slightly alkaline to litmus paper. The solution contains about 30 per cent of ammonium thioacetate.

TESTS OF PURITY

Non-volatile Matter. — 10 c.c. of ammonium thioacetate solution on evaporation and ignition should leave no weighable residue. (Indicating less than 0.005 per cent.)

Ammonium Carbonate. — On adding 3 c.c. of calcium chloride solution to 10 c.c. of ammonium thioacetate solution, no precipitate should form, even on warming. (Indicating less than 0.003 per cent $[NH_4]_2CO_3$.)

Sulphates. — A mixture of 10 c.c. of ammonium thioacetate solution with 10 c.c. of diluted acetic acid should not immediately be rendered turbid on the addition of barium chloride solution. (Indicating less than 0.0002 per cent SO_3.)

NOTE. — The reagent can be kept only a short time unchanged; it readily becomes turbid. It is, therefore, advantageous to prepare only sufficient for eight to ten days' use, which may be done by dissolving the thioacetic acid in a slight excess of ammonia water.

Regarding the use of ammonium thioacetate solution in analysis, see R.

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Schiff and N. P. Tarugi, Ber., **27**, 3437 (1894) [or Z. anal. Chem., **34**, 456 (1895)]; *abst.*, J. Chem. Soc., **68**, II, 84 (1895). R. Schiff, Ber., **28**, 1204 (1895); *abst.*, J. Chem. Soc., **68**, II, 370 (1895).

ANILINE

$C_6H_5NH_2$. Mol. Wt. 93.07

A colorless, oily, strongly refractive liquid, which rapidly becomes brown on exposure to light and air.

Aniline is soluble in about 35 parts of water. It solidifies in a freezing mixture, and then melts at −8° C. Its specific gravity is 1.027; its boiling-point, 183° C.

TEST OF PURITY

Hydrocarbons and Nitrobenzene. — 5 c.c. of aniline dissolved in 10 c.c. of hydrochloric acid should form a clear fluid which, on being diluted with 15 c.c. of water, should not become cloudy on cooling. (Indicating none present.)

NOTE. — Regarding the examination of aniline, see Lunge, Chem.-tech. Untersuch.-Meth., 5 ed., **3**, 886, 890 (1905); compare Watt's Dict. of Chem., **1**, 271 (1894). G. Schultz, Chemie des Steinkohlenteers, **1**, 289 (1886); compare Thorpe's Dict. of Appld. Chem., 2 ed., **1**, 162 (1895).

ANTIMONY OXIDE

(Antimonous Oxide; Antimony Trioxide)

Sb_2O_3. Mol. Wt. 288.4

A white powder, insoluble in water, but soluble in hydrochloric acid, tartaric acid, alkali bitartrates, and in solutions of potassium or sodium hydroxide. Antimony trioxide is neutral to litmus paper.

TESTS OF PURITY

Arsenic. — On dissolving 1 gm. of antimony trioxide in 3 c.c. of hydrochloric acid (sp.gr. 1.19), and adding 3 c.c. of stannous chloride solution, the mixture should not darken within one hour. (Indicating less than 0.0015 per cent As.)

Foreign Heavy Metals. — Dissolve 1 gm. of antimony trioxide with the aid of heat in 30 c.c. of sodium hydroxide solution (sp.gr. 1.3). Dilute the solution with 20 c.c. of water and add hydrogen sulphide water. Neither a white nor a brownish-black precipitate should form. (Indicating none present.)

Chlorides. — Dissolve 1 gm. of antimony trioxide with the aid of heat in 30 c.c. of sodium hydroxide solution (sp.gr. 1.3). Add to the solution 70 c.c. of nitric acid, filter, and to the filtrate add silver nitrate solution. The liquid may exhibit at most a slight turbidity, but no precipitate should form. (Indicating less than 0.05 per cent Cl.)

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ARSENIC TRIOXIDE

(Arsenous Acid; [Acid] Arsenous Anhydride)

As_2O_3. Mol. Wt. 197.92

White, vitreous (amorphous), or porcelain-like crystalline pieces, or white powder. The amorphous acid is more easily and quickly soluble than the crystalline, but the saturated solution of the former is not permanent, the less soluble crystalline form being gradually deposited from it. The crystalline acid is very slowly soluble in about 65 parts of cold water, and quickly soluble in 15 parts of boiling water.

TESTS OF PURITY

Non-volatile Matter. — 1 gm. of arsenic trioxide cautiously heated in a porcelain dish (use a hood!) should completely volatilize and leave no weighable residue. (Indicating less than 0.05 per cent.)

Barium Sulphate, Talcum, Calcium Sulphate, etc. — 0.5 gm. of arsenic trioxide should be perfectly soluble in a mixture of 5 c.c. of ammonia water and 5 c.c. of water, and should yield a clear solution. (Indicating none present.)

Arsenic Sulphide. — Dissolve 5 gm. of arsenic trioxide in a mixture of 5 c.c. of sodium hydroxide solution and 15 c.c. of water; on adding to the clear solution 2 drops of lead acetate solution, no change should appear. (Indicating less than 0.0005 per cent S.)

Quantitative Determination. — Dissolve* 1 gm. of arsenic trioxide with the aid of 6 gm. of potassium bicarbonate in 50 c.c. of boiling water, then allow to cool to 15° C., and add sufficient water to make the whole measure 100 c.c. Dilute 10 c.c. of this solution with 50 c.c. of water, and titrate with decinormal iodine solution.

1 c.c. of decinormal I = 0.004948 gm. of As_2O_3, log. 69443.

ASBESTOS PREPARATIONS

COPPER OXIDE ASBESTOS—SILVER ASBESTOS

These two forms of asbestos are prepared according to the method of Pregl,† and especially intended for use in elementary analysis.

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* It may be quicker to dissolve 1 gm. in a freshly prepared solution of sodium hydroxide (sulphur-free), slightly acidulate with hydrochloric acid, and then add the 6 gm. of bicarbonate. No heat is necessary in this case. It is always quicker to use a powdered sample. Regarding the use of arsenic trioxide in analysis see Mohr's Lehrb. Chem.-anal. Titriermeth., 7 ed., p. 389 ff. (1896); Sutton's Volumetric Analysis, 10 ed., 139 (1911).

† F. Pregl, Ber., **38**, 1434 (1905); *abst.*, J. Chem. Soc., **88**, II, 420 (1905).

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AZOLITMIN

Azolitmin is a particularly pure, water-soluble coloring matter made from litmus. It occurs in the form of blackish-violet scales.

Azolitmin is used as an indicator, usually in 1 per cent solution. To make this, dissolve 1 gm. of azolitmin in 80 c.c. of water with the aid of heat, then add 20 c.c. alcohol (about 85 per cent by weight), and filter the solution when cold.

TEST OF SENSITIVENESS

Add 0.1 c.c. of the above azolitmin solution to 50 c.c. of distilled water, free from carbon dioxide. The bluish-red color of the liquid should be changed to red by the addition of, at most, 0.05 c.c. of decinormal hydrochloric acid, and must be changed to bluish-violet by the addition of, at most, 0.05 c.c. of decinormal potassium hydroxide.

BARIUM ACETATE

$Ba(C_2H_3O_2)_2+H_2O$. Mol. Wt. 273.43

A white, crystalline powder, soluble in 2 parts of water and in about 100 parts of alcohol.

TESTS OF PURITY

Chlorides. — The solution of 1 gm. of barium acetate in 20 c.c. of water, acidulated with 5 c.c. of nitric acid, must show at most a slight opalescence on the addition of silver nitrate solution. (Indicating less than 0.002 per cent Cl.)

Calcium and Alkalies. — Dissolve 5 gm. of barium acetate in 200 c.c. of water, add to the solution 2 c.c. of hydrochloric acid, and heat to boiling. Now add 15 c.c. of 16 per cent sulphuric acid, allow to stand fifteen hours, filter, and mix the filtrate with alcohol (about 85 per cent by weight.) At most a faint opalescence should be observed; and on evaporating in a platinum dish and igniting, not more than 0.004 gm. of residue should remain. (Indicating not more than 0.08 per cent.)

Heavy Metals. — The 1 : 20 aqueous solution should not appear changed on the addition of hydrogen sulphide water; ammonia water with ammonium sulphide solution should produce no dark color and no precipitate. (Indicating none present.)

Nitrates. — The blue color imparted by 1 drop of 1 : 1000 indigo solution to a solution of 1 gm. of barium acetate and a small crystal of sodium chloride in 10 c.c. of water should not disappear on the addition of 10 c.c. of concentrated sulphuric acid followed by shaking. (Indicating less than 0.0032 per cent N_2O_5.)

BARIUM CARBONATE

$BaCO_3$. Mol. Wt. 197.37

White pieces or powder, almost insoluble in water free from carbon dioxide.

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TESTS OF PURITY

Solubility in Dilute Hydrochloric Acid. — 5 gm. of barium carbonate should be completely soluble in 10 c.c. of hydrochloric acid diluted with 50 c.c. of water.

Barium Hydroxide. — If 1 gm. of barium carbonate is shaken repeatedly during half an hour with 20 c.c. of water free from carbon dioxide, the solution after filtering should not be alkaline, and after evaporation and ignition not more than 0.002 gm. of residue should remain. (Indicating not more than 0.223 per cent $Ba[OH]_2$).

Calcium and Alkalies. — Dissolve 5 gm. of barium carbonate in 10 c.c. of hydrochloric acid and 200 c.c. of water, and heat the solution to boiling. Then add 15 c.c. of 16 per cent sulphuric acid, allow to stand fifteen hours, filter, and mix the filtrate with alcohol (about 85 per cent by weight). Not more than a faint opalescence should be visible, and on evaporating in a platinum dish and igniting, not more than 0.003 gm. of residue should remain. (Indicating not more than 0.06 per cent.)

Heavy Metals. — Neither hydrogen sulphide water, nor ammonia water with ammonium sulphide solution, should produce a dark color or a precipitate on being added to a solution of 1 gm. of barium carbonate in 5 c.c. of hydrochloric acid and 15 c.c. of water. The solution of barium carbonate in acid and water must be previously boiled to free it from carbon dioxide. (Indicating none present.)

Chlorides. — The solution of 1 gm. of barium carbonate in 5 c.c. of nitric acid and 25 c.c. of water should not be affected by silver nitrate solution. (Indicating less than 0.0005 per cent Cl.)

Nitrates. — The blue color imparted by 1 drop of a 1 : 1000 indigo solution to the solution of 1 gm. of barium carbonate and a small crystal of sodium chloride in 10 c.c. of diluted acetic acid should not disappear on the addition of 10 c.c. of concentrated sulphuric acid followed by shaking. (Indicating less than 0.0032 per cent N_2O_5.)

Quantitative Determination. — Dissolve 1 gm. of barium carbonate in a mixture of 15 c.c. of normal solution of hydrochloric acid and 50 c.c. of water, and titrate the solution with normal potassium hydroxide solution, using methyl orange as indicator.

1 c.c. of normal HCl = 0.098685 gm. of $BaCO_3$, log. 99425.

BARIUM CHLORIDE

$BaCl_2+2H_2O$. Mol. Wt. 244.32

Colorless crystals, soluble in 2.5 parts of cold, and in 1.5 parts of hot, water, and insoluble in absolute alcohol. The aqueous solution is neutral to litmus paper.

TESTS OF PURITY

Alkalies. — Dissolve 3 gm. of barium chloride in 100 c.c. of water, add 2 c.c. of hydrochloric acid and heat to boiling; then add 10 c.c. of 16 per cent sulphuric acid, allow to stand fifteen hours, and filter. On evaporating the filtrate in a platinum dish and igniting, any residue remaining should weigh not more than 0.001 gm. (Indicating not more than 0.0333 per cent.)

Strontium and Calcium Chlorides. — On shaking 1 gm. of powdered barium chloride with 20 c.c. of absolute alcohol five minutes, and then filtering, the filtrate on being evaporated and ignited should leave no weighable residue. (Indicating less than 0.025 per cent.)

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Heavy Metals. — 20 c.c. of the 1 : 20 aqueous solution should not afford a dark coloration or a precipitate when hydrogen sulphide water is added; nor when ammonia water and ammonium sulphide solution are added. (Indicating none present.)

Nitrates. — On adding 1 drop of a 1 : 1000 indigo solution to a solution of 1 gm. of barium chloride in 10 c.c. of water, the blue color must not disappear on the addition of 10 c.c. of concentrated sulphuric acid followed by shaking. (Indicating less than 0.0032 per cent N_2O_5.)

Chlorates. — On warming 2 gm. of powdered barium chloride with 10 c.c. of concentrated hydrochloric acid in a test-tube, neither the crystals nor the liquid should acquire a yellow color; nor should the odor of chlorine become perceptible. (Indicating less than 0.025 per cent Cl_2O_5.)

BARIUM DIOXIDE

(Barium Super- or Peroxide)

BaO_2. Mol. Wt. 169.37

White, or grayish-white powder, insoluble in water, but soluble in cold hydrochloric acid with the formation of hydrogen peroxide. The preparation should contain at least 82 per cent of BaO_2.

TEST OF PURITY

Quantitative Determination. — Introduce 1 gm. of barium peroxide into a graduated flask of 100 c.c. capacity, and add to it 5 gm. of potassium iodide dissolved in 30 c.c. of water. Add 10 c.c. of hydrochloric acid, and allow the mixture to stand in the stoppered flask for about half an hour, with frequent shaking. Then fill the flask to the mark, mix, and titrate 10 c.c. of the mixture with decinormal sodium thiosulphate solution, using starch solution as the indicator. At least 9.7 c.c. of decinormal sodium thiosulphate should be required.

1 c.c. of decinormal $Na_2S_2O_3 = 0.0084685$ gm. of BaO_2, log. 92780.

BARIUM HYDROXIDE

(Barium Hydrate)

$Ba(OH)_2+8H_2O$. Mol. Wt. 315.51

White crystals, soluble in 20 parts of cold, and in 3 parts of boiling, water, a trace of barium carbonate almost invariably remaining undissolved. The aqueous solution is alkaline to litmus paper.

TESTS OF PURITY

Chlorides. — The solution of 1 gm. of barium hydroxide in 5 c.c. of nitric acid and 25 c.c. of water should not be affected by silver nitrate solution. (Indicating less than 0.0005 per cent Cl.)

Calcium and Alkalies. — Dissolve 3 gm. of barium hydroxide in 100 c.c. of water, add 5 c.c. of hydrochloric acid, and heat to boiling. Then add 10 c.c. of diluted sulphuric acid, allow to stand fifteen hours, and filter. On evaporating

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the filtrate in a platinum dish and igniting, a residue weighing more than 0.002 gm. should not remain. (Indicating not more than 0.0667 per cent.)

Heavy Metals. — The solution of 1 gm. of barium hydroxide dissolved in 20 c.c. of water should not acquire a dark color, or yield a precipitate, on the addition of 2 c.c. hydrochloric acid followed by hydrogen sulphide water; nor should a similar solution do so on adding ammonia water to alkaline reaction, followed by ammonium sulphide solution. (Indicating none present.)

Sulphides. — The 1 : 20 aqueous solution of barium hydroxide, when acidulated with hydrochloric acid, should not have an odor of hydrogen sulphide, nor should it acquire a dark color on adding lead acetate solution. (Indicating less than 0.0027 per cent S.)

Quantitative Determination. — Dissolve 1 gm. of barium hydroxide in 100 c.c. of water, add 1 drop of methyl orange solution, and titrate with normal hydrochloric acid. Not less than 6.3 c.c. of the normal acid should be necessary to produce the red end-point.

1 c.c. of normal HCl = 0.157755 gm. of $Ba(OH)_2+8H_2O$, log. 19799.

BARIUM HYDROXIDE SOLUTION

(Baryta Water)

A clear, colorless liquid, of strongly alkaline reaction. Baryta water contains 3.3 per cent of crystallized barium hydroxide, $Ba(OH)_2+8H_2O$.

TESTS OF PURITY

The tests to be made are those given under Barium Hydroxide. But for each gram of crystallized barium hydroxide, 30 c.c. of baryta water are to be taken. The quantitative determination is made as follows:

Quantitative Determination. — Titrate 50 c.c. of baryta water with normal solution of hydrochloric acid, using methyl orange as the indicator. At least 10.5 c.c. of normal solution of hydrochloric acid should be required to produce the red end-point.

1 c.c. of normal HCl = 0.157755 gm. of $Ba(OH)_2+8H_2O$, log. 19799.

BARIUM NITRATE

$Ba(NO_3)_2$. Mol. Wt. 261.39

Colorless crystals, soluble in 20 parts of cold, and in 2.8 parts of boiling, water. The aqueous solution is neutral to litmus paper. Barium nitrate is insoluble in absolute alcohol.

TESTS OF PURITY

Chlorides. — The 1 : 20 aqueous solution acidulated with nitric acid should show no change on the addition of silver nitrate solution. (Indicating less than 0.0005 per cent Cl.)

Alkalies and Heavy Metals. — The tests are to be carried out as described under Barium Chloride. In the test for alkalies do not use a platinum dish. 0.002 gm. residue is permissible. (Indicating not more than 0.0667 per cent alkalies.)

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BARIUM SULPHIDE

Gray, hard, amorphous pieces, prepared by fusing a mixture of heavy-spar, powdered coke, and sodium chloride. The preparation contains at least 50 per cent of barium sulphide (BaS), and is prepared in cube form for generation of hydrogen sulphide.

On allowing hydrochloric acid to flow over barium sulphide, a uniform current of hydrogen sulphide should be generated.

TESTS OF PURITY

Arsenic. — Heat 100 c.c. of nitric acid (sp.gr. 1.3) in a porcelain dish to 70° to 80° C., and add 10 gm. of finely powdered barium sulphide, gradually, in quantities of 0.2 to 0.3 gm. at a time; when all has been added, heat to boiling. Then add to the mixture 100 c.c. of arsenic-free, dilute sulphuric acid (1 : 5), evaporate on the water-bath as far as possible; continue to heat on the sand-bath until vapors of sulphuric acid begin to be evolved; cool, and finally stir the cooled residue with 100 c.c. of water.

Set a Marsh apparatus in operation, using 20 gm. of arsenic-free, granulated zinc and dilute (1 : 5) sulphuric acid, and then introduce the above residue suspended in water, in small quantities at a time, into the generating flask of the Marsh apparatus. No deposit of arsenic should be observable in the reduction tube after the apparatus has been in operation one hour. (Indicating less than 0.0001 per cent As.)

Quantitative Determination. — Dissolve 0.73 gm. of copper sulphate ($CuSO_4 + 5H_2O$) in 50 c.c. of water, and then add to it 1 gm. of powdered barium sulphide, representing the average quality of the goods. To this add, while stirring, 10 c.c. of acetic acid (sp.gr. 1.040–1.042) by drops. Filter, expel the hydrogen sulphide from the filtrate by boiling, and add an excess of ammonia water. No blue color should appear. (Indicating at least 50 per cent BaS.)

NOTE. — This chemical serves for the preparation of arsenic-free hydrogen sulphide. The hydrochloric acid to be used for this purpose is to be tested for arsenic.

BENZENE

(Benzol)

C_6H_6. Mol. Wt. 78.05

A clear, colorless liquid, of characteristic odor, insoluble in water, but easily soluble in alcohol and in ether. Benzene upon cooling forms rhombic, crystalline scales, which melt at +4° C. Its specific gravity is 0.883, and it boils at 79–80° C.

TESTS OF PURITY

Thiophene.* — On shaking 50 c.c. of benzene with 20 c.c. of concentrated sulphuric acid, the sulphuric acid should remain colorless; on now adding a

* See C. Schwalbe: "The Sulphur Content of Pure Benzenes"; Z. Farb.-Textl. Ind., **3**, 461 (1904); *abst.*, J. Chem. Soc., 88, 1, 124 (1905). Z. Farb.-Textl. Ind., **4**, 113 (1905); *abst.*, J. Soc. Chem. Ind., **24**, 271 (1905).

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crystal of isatin, and again shaking and allowing to stand one hour, the sulphuric acid should have neither a green nor a blue color. (Indicating none present.)

Carbon Disulphide.*— Thoroughly mix 50 c.c. of benzene with 50 c.c. of alcoholic potassium hydroxide solution (11 gm. of potassium hydroxide in 90 gm. of absolute alcohol), and allow the mixture to stand several hours at a temperature of about 20° C. Shake with about 100 c.c. of water, remove the aqueous solution from the benzene, neutralize the aqueous portion with acetic acid, and add copper sulphate solution. No precipitate should form. (Indicating less than 0.0072 per cent CS_2.)

BENZIDINE

(Paradiaminodiphenyl)

$(C_6H_4)_2(NH_2)_2$. **Mol. Wt. 184.12**

A white or slightly reddish crystalline powder, melting at 128° C. Benzidine is very difficultly soluble in cold water, but more readily in boiling water, in alcohol, and in ether.

TEST OF PURITY

Sulphates. — On adding barium chloride to a solution of 1 gm. of benzidine in 50 c.c. of water acidulated with 5 c.c. of hydrochloric acid, no precipitate of barium sulphate should form on standing fifteen hours. (Indicating less than 0.0125 per cent SO_3.)

Non-volatile Matter. — 1 gm. of benzidine upon ignition should leave no weighable residue. (Indicating less than 0.05 per cent.)

NOTE. — Regarding the use of benzidine in the determination of sulphuric acid see Wolf J. Müller and K. Dürkes, Z. anal. Chem. **42**, 477 (1903); *abst.*, J. Chem. Soc., **84**, II, 751 (1903). W. J. Müller, Z. angew. Chem., **16**, 653, 1017 (1903); *abst.*, J. Chem. Soc., **86**, II, 83 (1904). Raschig, Z. angew. Chem., **16**, 617 (1903); *abst.*, J. Chem. Soc., **84**, II, 572 (1903). Z. angew. Chem., **16**, 818 (1903); *abst.*, J. Chem. Soc., **84**, II, 691 (1903). G. Knorre, J. Soc. Chem. Ind., **28**, 2 (1905); *abst.*, J. Chem. Soc., **88**, II, 351 (1905). Friedheim and Nydegger, Z. angew. Chem., **20**, 9 (1907); *abst.*, J. Chem. Soc., **92**, II, 196 (1907). G. Knorre, Chem. Ztg., **34**, 405 (1910); *abst.*, J. Chem. Soc., **98**, II, 545 (1910).

BENZIDINE FOR BLOOD TEST

$(C_6H_4)_2(NH_2)_2$. **Mol. Wt. 184.12**

A light-brown, crystalline powder, very difficultly soluble in cold water, but more easily soluble in boiling water, in alcohol, and in ether.

TEST OF PURITY

Test of Suitability for Blood Detection. — Dissolve 1 gm. of benzidine in 10 c.c. of concentrated acetic acid (sp.gr. 1.064), and to 1 c.c. of this solution

* F. Frank: Chem. Ind., **24**, 237, 262 (1901) [or Chem. Zentr., (5) **5**, 1, 1251 (1901)]; *abst.*, J. Soc. Chem. Ind., **20**, 566 (1901).

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add 10 c.c. of 3 per cent solution of hydrogen peroxide.* No color at all, or at most a very faint color should appear within a few minutes. A small quantity of blood added to this solution produces a blue color.

NOTE. — Regarding the use of benzidine for detection of blood see O. and R. Adler, Z. physiol. Chem., **41**, 59 (1904); *abst.*, J. Chem. Soc., **86**, II, 459 (1904). O. Schumm and C. Westphal, Z. physiol. Chem., **46**, 510 (1905); *abst.*, J. Chem. Soc., **90**, II, 207 (1906). E. Walter, Deut. med. Wochenschr., **35**, I, 130 (1909). Th. Messerschmidt, Münch. med. Wochenschr., **56**, 388 (1909); *abst.*, J. Am. Med. Assoc., **52**, 1150 (1909). A. Ascarelli, Deut. med. Wochenschr., **34**, II, 2307 (1908); *abst.*, C. A., **3**, I, 764 (1909). F. W. White, Boston Med. and Surg. Jour., **160**, 733 (1909). H. Merkel, Münch. med. Wochenschr., **56**, II, 2358 (1909), *abst.*, C. A., **4**, I, 334 (1910). O. Schumm, Arch. d. Pharm., **247**, 16 (1909); *abst.*, J. Chem. Soc., **96**, II, 195 (1909). E. Schlesinger and F. Holst, Deut. med. Wochenschr., **32**, II, 1444 (1906); *abst.*, J. Am. Med. Assoc., **47**, 1335 (1906). O. Schumm, Deut. med. Wochenschr., **33**, II, 1471 (1907); *abst.*, J. Chem. Soc., **92**, II, 827 (1907). E. Walter, Deut. med. Wochenschr., **36**, I, 309 (1910); *abst.*, C. A., **4**, 1184 (1910). M. Einhorn, Deut. med. Wochenschr., **33**, II, 1089 (1907); *abst.*, J. Am. Med. Assoc., **48**, 2161 (1907), and Med. Record, June 8 (1907). Utz, Chem. Ztg., **31**, 737 (1907); *abst.*, J. Chem. Soc., **92**, II, 916 (1907).

BENZIN

(Petroleum Ether; Petroleum Benzin)

A colorless, non-fluorescent, very inflammable liquid consisting of the low-boiling portions of petroleum, and having a characteristic but not unpleasant odor. Specific gravity 0.666 to 0.686. Benzin does not solidify at 0° C. If 100 c.c. are heated on the water-bath at least 80 c.c. will distill between 40° and 70° C.

TESTS OF PURITY†

Non-volatile Matter and Heavy Oils. — 20 c.c. of benzin warmed on the water-bath should volatilize and leave no residue. When dropped upon paper, and allowed to evaporate, it should leave no greasy spot. (Indicating none present.)

Acids. — 10 c.c. of benzin shaken with 10 c.c. of water should not impart to the water an acid reaction. (Indicating none present.)

Sulphur Compounds and Reducing Substances. — A mixture of 5 c.c. of benzin and 10 c.c. of alcoholic ammoniacal silver nitrate solution‡ is heated on a water-bath protected from the light. No brown color should appear within five minutes. (Indicating none present.)

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*1 c.c. perhydrol + 9 c.c. water.

† The testing of benzin for benzene by nitrating the latter with nitro-sulphuric acid was not adopted here, because it is impossible to obtain a benzin that will stand this test.

‡ 10 c.c. of a solution of 1 gm. of silver nitrate in 100 c.c. of alcohol (about 85 per cent by weight) are mixed with 5 c.c. of an alcoholic solution of ammonia (9–10 per cent NH_3.).

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BISMUTH SUBNITRATE

(Bismuth Basic Nitrate)

A white, microcrystalline powder, insoluble in water and in alcohol, but soluble in diluted hydrochloric, nitric, or sulphuric acid. It contains 70.8–73.5 per cent bismuth, Bi. On shaking bismuth subnitrate with water, it imparts an acid reaction to the latter.

TESTS OF PURITY

Carbonates, Lead, Copper, Salts of the Alkalies, etc. — 0.5 gm. of bismuth subnitrate should completely dissolve in 25 c.c. of cold 16 per cent sulphuric acid without the evolution of carbon dioxide (indicating no carbonate present), and should yield a clear solution (indicating less than 0.165 per cent Pb). 10 c.c. of this solution, with an excess of ammonia water, should yield a colorless filtrate. (Indicating less than 0.01 per cent Cu.) Another 10 c.c. of this solution, diluted with 100 c.c. of water and treated with hydrogen sulphide to precipitate completely the bismuth, should yield a filtrate leaving no weighable residue on evaporation and ignition. (Indicating less than 0.25 per cent alkali salts.)

Chlorides. — On adding silver nitrate solution to a solution of 0.5 gm. of bismuth subnitrate in 5 c.c. of nitric acid, not more than a slight opalescent turbidity should develop. (Indicating less than 0.01 per cent Cl.)

Sulphates. — The solution of 0.5 gm. of bismuth subnitrate in 5 c.c. of nitric acid should show no change on the addition of 5 c.c. of water followed by 5 to 10 drops of barium nitrate solution. (Indicating less than 1.5 per cent SO_3.)

Ammonia. — On warming 1 gm. of bismuth subnitrate with 10 c.c. of sodium hydroxide solution (sp.gr. 1.3), no ammonia should be evolved (to be ascertained by moistened litmus paper). (Indicating less than 0.0035 per cent NH_3.)

Residue on Ignition. — On igniting 1 gm. of bismuth subnitrate, there should remain a residue of bismuth oxide weighing 0.79 to 0.82 gm. (Indicating 79 to 82 per cent.)

Arsenic. — The residue of bismuth oxide obtained above is triturated to a fine powder, and dissolved in sulphuric acid with heat. Set a Marsh apparatus in operation, using 20 gm. of arsenic-free, granulated zinc, and dilute (1 : 5) sulphuric acid, then introduce the bismuth solution into the evolution flask. No deposit of arsenic should be visible in the reduction tube within half an hour. (Indicating less than 0.001 per cent As.)

BISMUTH AND POTASSIUM IODIDE SOLUTION

An aqueous solution containing in each kilo 25 gm. of bismuth iodide and 100 gm. of potassium iodide.

BROMINE

Br. Atomic Wt. 79.92

A dark-red to brown, almost black, liquid, of specific gravity 3.14, and boiling at about 63° C. Bromine dissolves in about 30

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parts of water. It is easily soluble in alcohol, ether, chloroform, and carbon disulphide.

TESTS OF PURITY

Non-volatile Matter. — 5 gm. of bromine warmed in a porcelain dish on the water-bath should volatilize and leave no weighable residue. (Indicating less than 0.01 per cent.)

Sulphuric Acid and Organic Bromine Compounds (Bromoform; Carbon Tetrabromide). — 2 gm. of bromine should dissolve clear in a mixture of 10 c.c. of solution of sodium hydroxide (sp.gr. 1.168–1.172), and 10 c.c. of water. After standing six hours no oily drops should be visible in this solution. (Indicating no organic bromine compounds present.) If the solution is then acidulated with hydrochloric acid and boiled until the bromine has volatilized, the addition of barium chloride solution should cause no apparent change. (Indicating less than 0.005 per cent SO_3.)

Iodine. — Dissolve 1 gm. of bromine in 40 c.c. of water, add 4 gm. of powdered iron, and shake two to three minutes. Filter, add starch solution and ferric chloride solution to the filtrate. No blue color should appear. (Indicating less than 0.75 per cent I.)

Determination of Chlorine Content. — Tare a graduated 250 c.c. flask containing 15 gm. of potassium iodide and 30 c.c. of water. Add about 1 c.c. of water-free bromine and weigh. After complete solution of the iodine, dilute to the mark, mix, and titrate 25 c.c. of the liquid with decinormal solution of sodium thiosulphate. If A is the amount of bromine weighed off, and I the amount of iodine liberated, and X the amount of chlorine in A, then $X = \frac{I - 1.588A}{1.9912}$. The bromine is $A - X$.

1 c.c. of decinormal $Na_2S_2O_3 = 0.012692$ gm. of I, log. 10352.

BROMINE WATER

A saturated, aqueous solution, containing about 3 per cent of bromine, Br.

TESTS OF PURITY

Sulphuric Acid. — Add 1 c.c. of hydrochloric acid and some barium chloride solution to 50 c.c. of bromine water, and boil the liquid until the bromine has been completely expelled from it. No precipitate of barium sulphate should separate on standing. (Indicating less than 0.00002 per cent SO_3.)

Bromine Content. — Let 10 c.c. of bromine water run into a solution of 5 gm. of potassium iodide in 100 c.c. of water, allow to stand half an hour in a stoppered flask, and then titrate with decinormal sodium thiosulphate solution.

1 c.c. of decinormal $Na_2S_2O_3 = 0.007992$ gm. of Br., log. 9026$\bar{6}$.

BRUCINE

$C_{23}H_{26}N_2O_4 + 2H_2O$.* Mol. Wt. 430.36

Small, white crystals, difficultly soluble in cold, but more readily soluble in boiling, water. Brucine is easily soluble in alcohol (85

* Brucine also crystallizes with 4 molecules of water; then it dissolves in 320 parts of cold, and in 150 parts of boiling, water.

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per cent by weight), and in chloroform. The aqueous solution of brucine is alkaline to litmus paper, and is levogyrate. When dried at 100° C., brucine melts at 178° C.

TESTS OF PURITY

Water of Crystallization. — 1 gm. of brucine dried at 100° C. to constant weight should lose not more than 0.084 gm. (Indicating not more than 8.4 per cent.)

Nitric Acid. — 0.01 gm. of brucine should dissolve in 5 c.c. of pure concentrated sulphuric acid, and the solution should be colorless or, at most, of a scarcely perceptible faint pink color.* (Indicating less than 0.05 per cent N_2O_5.)

Quantitative Determination. — Dissolve 0.25 gm. of brucine in 50 c.c. of alcohol (85 per cent by weight), and titrate with decinormal hydrochloric acid, using lacmoid as the indicator. At least 5.8 c.c. of decinormal acid should be required to produce the red end-point.

1 c.c. of decinormal HCl = 0.043026 gm. of $C_{23}H_{26}N_2O_4+2H_2O$, log. 63373.

CADMIUM BOROTUNGSTATE SOLUTION

A perfectly clear, yellowish, or light brown, liquid, having a specific gravity of 3.28.

CADMIUM AND POTASSIUM IODIDE

(Potassium-Cadmium Iodide)

A white powder, easily soluble in water, and in alcohol. Cadmium and potassium iodide readily acquires a slight yellowish color on keeping.

TESTS OF PURITY

Foreign Metals. — (*a*) Dissolve 1 gm. of cadmium and potassium iodide in 20 c.c. of water, add to the solution 2 c.c. of potassium hydroxide solution (sp.gr. 1.138–1.140), and filter. Neither before nor after acidulating with hydrochloric acid should the filtrate yield a precipitate with hydrogen sulphide water. (Indicating none present.)

(*b*) Dissolve 1 gm. of cadmium and potassium iodide in 30 c.c. of water, and add to the solution 5 c.c. of ammonia water. The liquid should remain clear and colorless on shaking. (Indicating none present.)

Sulphates. — The 1 : 20 aqueous solution acidulated with hydrochloric acid should not be affected by barium chloride solution. (Indicating less than 0.01 per cent SO_3.)

Iodic Acid. — The 1 : 20 solution freshly prepared with boiled water should not immediately give a blue color on adding starch solution followed by 2 or 3 drops of dilute sulphuric acid. (Indicating less than 0.00125 per cent HIO_3.)

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* The sulphuric acid should be tested with diphenylamine to insure the absence of nitric acid.

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CALCIUM CARBONATE PRECIPITATED

$CaCO_3$. Mol. Wt. 100.07

A white, crystalline powder, insoluble in boiled water, but somewhat soluble in carbonated water.

TESTS OF PURITY

Solubility in Hydrochloric, Nitric, and Acetic Acids. — 5 gm. of calcium carbonate should be completely dissolved by 25 c.c. of hydrochloric acid, by 25 c.c. of nitric acid, and by 60 c.c. of 30 per cent acetic acid. The solutions must be clear and colorless.

Heavy Metals. — Dissolve 1 gm. of calcium carbonate in 5 c.c. of hydrochloric acid and 25 c.c. of water. This solution should not appear changed upon adding hydrogen sulphide water; then upon adding ammonia water in excess accompanied by 2 or 3 drops of ammonium sulphide solution neither a green color nor a precipitate should appear. (Indicating none present.)

Magnesium. — Dissolve 1 gm. of calcium carbonate in 5 c.c. of hydrochloric acid and 5 c.c. of water. To this solution add 10 c.c. of ammonia water and an excess of ammonium oxalate solution, allow to stand five hours, filter, and add to the filtrate sodium phosphate solution. No precipitate should form on standing fifteen hours. (Indicating less than 0.002 per cent Mg.)

Sulphates. — Dissolve 1 gm. of calcium carbonate in 5 c.c. of hydrochloric acid and 25 c.c. of water, boil the solution five minutes, and add barium chloride solution. No precipitate of barium sulphate should form on standing fifteen hours. (Indicating less than 0.0125 per cent SO_3.)

Chlorides. — 1 gm. of calcium carbonate dissolved in 5 c.c. of nitric acid and 25 c.c. of water, must show no change on the addition of silver nitrate solution. (Indicating less than 0.0005 per cent Cl.)

Phosphates. — Dissolve 10 gm. of calcium carbonate in 50 c.c. of nitric acid, add 25 c.c. of ammonium molybdate solution, and allow to stand fifteen hours at a temperature of 30° to 40° C. No yellow precipitate should form. (Indicating less than 0.001 per cent P_2O_5.)

Alkalies and Calcium Oxide. — Boil 5 gm. of calcium carbonate with 50 c.c. of water and then filter. The filtrate should not be alkaline to litmus, and upon evaporation followed by ignition should leave a residue weighing at most 0.001 gm. (Indicating not more than 0.02 per cent.)

CALCIUM CHLORIDE CRYSTALS

$CaCl_2+6H_2O$. Mol. Wt. 219.09

Colorless crystals, deliquescent in the air, and easily soluble in water and in alcohol. The 1 : 10 solution should be neutral to litmus paper.

TESTS OF PURITY

Substances Insoluble in Absolute Alcohol. — 2 gm. of calcium chloride should completely dissolve in 20 c.c. of absolute alcohol. (Indicating none present.)

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Heavy Metals. — 20 c.c. of the 1 : 10 aqueous solution should show no change on adding 1 c.c. of hydrochloric acid and hydrogen sulphide water. On further adding 5 c.c. of ammonia water and 2 to 3 drops of ammonium sulphide solution, no green color should develop, nor should a precipitate form. (Indicating none present.)

Sulphates. — On adding barium chloride solution to the solution of 2 gm. of calcium chloride in 20 c.c. of water acidulated with 1 c.c. of hydrochloric acid, no precipitate should form on standing fifteen hours. (Indicating less than 0.0038 per cent SO_3.)

Ammonium Salts. — On boiling 2 gm. of calcium chloride with 10 c.c. of sodium hydroxide solution, no ammonia should be evolved (to be ascertained by moistened litmus paper). (Indicating less than 0.00175 per cent NH_3.)

Barium. — On adding 20 c.c. of calcium sulphate solution to the solution of 2 gm. of calcium chloride in 20 c.c. of water, no change should appear within three hours. (Indicating less than 0.002 per cent Ba.)

Arsenic. — Introduce 20 gm. of arsenic-free, granulated metallic zinc into the generating flask of a Marsh apparatus, and start the hydrogen with dilute (1 : 5) sulphuric acid. Dissolve 5 gm. of calcium chloride in 20 c.c. of water, introduce the solution into the Marsh apparatus, and maintain the stream of gas for one hour. No deposit of arsenic should be visible in the reduction tube within this time. (Indicating less than 0.0002 per cent As.)

Nitrates. — A solution of 1 gm. of calcium chloride in 10 c.c. of water is mixed with one drop of indigo solution (1 : 1000) and 10 c.c. of concentrated sulphuric acid. (Caution! Hydrochloric acid is freely evolved!) The blue color of the solution should not disappear upon shaking. (Indicating less than 0.0032 per cent N_2O_5.)

CALCIUM CHLORIDE, DRY, GRANULATED

$CaCl_2+2H_2O$. Mol. Wt. 147.02

White, granular, porous masses in pieces the size of peas. It may be used for drying gases.

TESTS OF PURITY

Neutrality. — The solution of 10 gm. of calcium chloride in 100 c.c. of water must not turn phenolphthalein red. At most a very slight red may occur. Any red color must be discharged by the addition of 0.1 c.c. of decinormal hydrochloric acid. (Indicating not more than 0.0028 per cent CaO.)

Arsenic. — The tests to be carried out and conditions to be observed are those given under Calcium Chloride Crystals. (Indicating less than 0.0002 per cent As.)

CALCIUM CHLORIDE, FUSED

White crystalline masses, the formula of which is practically $CaCl_2$. Fused calcium chloride is chiefly used for drying liquids, e.g., ethers, esters, ethereal oils, hydrocarbons, etc.

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CALCIUM HYDROXIDE

(Slaked Lime)

$Ca(OH)_2$. Mol. Wt. 74.09

A white, dry powder.

The article is tested as to purity by the methods detailed under Calcium Oxide, from Marble.

CALCIUM OXIDE, FROM ICELAND SPAR

CaO. Mol. Wt. 56.07

White pieces in the form of Iceland spar crystals.

TESTS OF PURITY

Carbonates, Silicates, and Sulphates. — 3 gm. of calcium oxide slaked with 10 c.c. of water should completely dissolve in 15 c.c. of hydrochloric acid without effervescence. (Indicating less than 0.7 per cent CO_2, and no silicates present.) Dilute the solution with 50 c.c. of water, heat to boiling, and add barium chloride solution. On standing fifteen hours, not more than a scarcely perceptible, unweighable trace of barium sulphate should be present. (Indicating less than 0.017 per cent SO_3.)

Phosphates. — Dissolve 3 gm. of calcium oxide in 10 c.c. of water, mixed with 25 c.c. of nitric acid, and add 25 c.c. of ammonium molybdate solution. No yellow precipitate should form on standing fifteen hours at about 40° C. (Indicating less than 0.0033 per cent P_2O_5.)

Chlorides. — Dissolve 3 gm. of calcium oxide in 10 c.c. of water mixed with 25 c.c. of nitric acid. The solution should show no change on the addition of silver nitrate solution. (Indicating less than 0.00015 per cent Cl.)

Iron. — Dissolve 1 gm. of calcium oxide in 10 c.c. of water mixed with 10 c.c. of hydrochloric acid. The solution should not immediately afford a blue color on adding 0.5 c.c. of potassium ferrocyanide solution. (Indicating less than 0.01 per cent Fe.)

CALCIUM OXIDE, FROM MARBLE

(Lime)

CaO. Mol. Wt. 56.07

White pieces of granular structure which develop much heat on being sprinkled with half their weight of water, and become converted into a fine, dust-like powder of slaked lime. With three or four parts of water the slaked lime yields a thick paste. Calcium oxide is soluble in about 800 parts of cold, and in 1300 parts of boiling, water.

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TESTS OF PURITY

Carbonates, Silica, Alumina, and Sulphates. — Slake 5 gm. of calcium oxide with 10 c.c. of water. The magma so obtained should almost completely dissolve in 30 c.c. of hydrochloric acid without strong effervescence. (Indicating less than 5 per cent CO_2.) Dilute the solution with 20 c.c. of water, and filter. The insoluble residue, after washing and ignition, should weigh at most 0.005 gm. (Indicating not more than 0.1 per cent SiO_2.) To 25 c.c. of the filtrate, add barium chloride solution; the liquid should show no change within ten minutes. (Indicating less than 0.03 per cent SO_3.) Supersaturate 25 c.c. of the filtrate with ammonia water; the liquid should not contain more than a slight flocculent precipitate. (Indicating less than 0.25 per cent Al.)

Chlorides. — Dissolve 1 gm. of calcium oxide in 10 c.c. of water, mixed with 10 c.c. of nitric acid, and add silver nitrate solution. The liquid should not be rendered more than slightly opalescent. (Indicating less than 0.002 per cent Cl.)

CALCIUM PHOSPHATE, DIBASIC

(Secondary Calcium Phosphate; Dicalcium Phosphate)

$CaHPO_4+2H_2O$. Mol. Wt. 172.15

A white, crystalline powder, easily soluble in hydrochloric, nitric, or phosphoric acid, without effervescence; it is difficultly soluble in water.

Dicalcium phosphate does not dissolve in boiling water without decomposition; it produces a liquid of acid reaction, and an amorphous residue the composition of which approximates that of the neutral salt.

TESTS OF PURITY

Arsenic. — The test is carried out as described under Calcium Phosphate, Monobasic, on page 64, using a solution of 2 gm. of dicalcium phosphate in 5 c.c. of hydrochloric acid and 15 c.c. of water. (Indicating less than 0.0005 per cent As.)

Chlorides. — Dissolve 1 gm. of dicalcium phosphate in 5 c.c. of nitric acid and 15 c.c. of water; on adding silver nitrate solution, not more than a slight opalescent turbidity should be visible. (Indicating less than 0.002 per cent Cl.)

Heavy Metals. — The solution of 1 gm. of dicalcium phosphate in 5 c.c. of hydrochloric acid and 15 c.c. of water should afford a pure white precipitate on the addition of hydrogen sulphide water and 10 c.c. of ammonia water. (Indicating none present.)

Sulphates. — On shaking 1 gm. of dicalcium phosphate with 20 c.c. of water and filtering, the filtrate, on adding 1 c.c. of hydrochloric acid and barium chloride solution, should develop no precipitate within fifteen hours. (Indicating less than 0.0075 per cent SO_3.)

Residue on Ignition. — On ignition, dicalcium phosphate should yield 74 to 75 per cent of its weight of residue.*

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* On ignition, dicalcium phosphate is converted into calcium pyrophosphate, whereby it theoretically loses 26.12 per cent of water.

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CALCIUM PHOSPHATE, MONOBASIC

(Calcium Biphosphate; Calcium Acid [or Super] Phosphate; Primary Calcium Phosphate; Monocalcium Phosphate)

$Ca(H_2PO_4)_2+H_2O$. Mol. Wt. 252.2

Colorless, pearly scales, which readily deliquesce in the air.

Calcium biphosphate dissolves in much water, apparently without decomposition; with a small quantity of water, an amorphous salt separates, richer in calcium, while the solution exhibits a strongly acid reaction.

On heating a 1 : 50 aqueous solution of calcium biphosphate to boiling, a precipitate forms.

TESTS OF PURITY

Arsenic. — Introduce 20 gm. of arsenic-free, granulated zinc into the generating flask of a Marsh apparatus, and start the stream of hydrogen by adding dilute (1 : 5) sulphuric acid. After the apparatus and reagents have been tested in the usual manner, dissolve 2 gm. of calcium biphosphate in 5 c.c. of hydrochloric acid and 15 c.c. of water. Introduce this solution into the Marsh apparatus, and maintain the flow of gas for one hour. No deposit of arsenic should be visible in the reduction tube within this time. (Indicating less than 0.0005 per cent As.)

Chlorides. — A solution of 1 gm. of calcium biphosphate in 5 c.c. of nitric acid and 15 c.c. of water should not be rendered more than slightly opalescent on the addition of silver nitrate solution. (Indicating less than 0.002 per cent Cl.)

Heavy Metals. — On adding hydrogen sulphide water and 10 c.c. of ammonia water to a solution of 1 gm. of calcium biphosphate in 5 c.c. of hydrochloric acid and 15 c.c. of water, a pure white precipitate should form. (Indicating none present.)

Sulphates. — 1 gm. of calcium biphosphate dissolved in 5 c.c. of hydrochloric acid and 20 c.c. of water should not be changed by the addition of barium chloride solution. (Indicating less than 0.0875 per cent SO_3.)

CALCIUM PHOSPHATE, TRIBASIC

(Tertiary Calcium Phosphate; Tricalcium Phosphate)

$Ca_3(PO_4)_2$. Mol. Wt. 310.29

A white, amorphous powder, insoluble in cold water. Tricalcium phosphate is gradually decomposed by boiling water into an insoluble basic salt and an easily soluble acid salt. It is easily soluble in hydrochloric or nitric acid, and without effervescence.

TESTS OF PURITY

Arsenic. — The test is carried out as detailed under Calcium Phosphate, Monobasic, see above, using a solution of 2 gm. of tricalcium phosphate in

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5 c.c. of hydrochloric acid and 15 c.c. of water. (Indicating less than 0.0005 per cent As.)

Sulphates. — Shake 1 gm. of tricalcium phosphate with 20 c.c. of water, filter, and add to the filtrate 1 c.c. of hydrochloric acid and barium chloride solution. No precipitate of barium sulphate should form on standing fifteen hours. (Indicating less than 0.0075 per cent SO_3.)

Chlorides. — The solution of 1 gm. of tricalcium phosphate in 5 c.c. of nitric acid and 15 c.c. of water should not show more than a slight opalescent turbidity on adding silver nitrate solution. (Indicating less than 0.002 per cent Cl.)

Heavy Metals. — The solution of 1 gm. of tricalcium phosphate in 5 c.c. of hydrochloric acid and 15 c.c. of water should yield a pure white precipitate on adding hydrogen sulphide water and 10 c.c. of ammonia water. (Indicating none present.)

CALCIUM SULPHATE

(Gypsum)

$CaSO_4+2H_2O$. Mol. Wt. 172.17

A fine white powder, difficultly soluble in water (about 1 : 500).

TESTS OF PURITY

Iron, Magnesium, and Alkalies. — On warming 2 gm. of calcium sulphate with 10 c.c. of hydrochloric acid and 100 c.c. of water, a clear solution should result, which, on the addition of 15 c.c. of ammonia water and a few drops of ammonium sulphide solution, should not acquire a greenish or dark color. (Indicating less than 0.00375 per cent Fe.) To the solution, no matter whether a precipitate of calcium sulphate has formed or not, add ammonium oxalate solution in slight excess, filter, evaporate the filtrate, and ignite in a platinum dish. There should not remain a residue weighing more than 0.002 gm. (Indicating not more than 0.1 per cent magnesium and alkalies.)

CALCIUM SULPHIDE

(Sulphurated Lime)

Bulky, light-gray cubes, from which dilute hydrochloric acid sets free copious quantities of hydrogen sulphide gas. The preparation contains at least 60 per cent calcium sulphide (CaS).

TESTS OF PURITY

Arsenic. — This is carried out as detailed under Barium Sulphide on page 54. (Indicating less than 0.0001 per cent As.)

Quantitative Determination. — Into a solution of 2.08 gm. of copper sulphate ($CuSO_4+5H_2O$) in 50 c.c. of water introduce 1 gm. of powdered calcium sulphide, and while stirring add by drops 10 c.c. of hydrochloric acid. Warm the mixture about fifteen minutes on the water-bath and then filter. The filtrate upon the addition of ammonia water in excess must show no blue color. (Indicating at least 60 per cent of CaS.)

NOTE. — This preparation serves for the generation of arsenic-free hydrogen sulphide. The hydrochloric acid used for this purpose should also be tested for arsenic.

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CARBON DISULPHIDE

CS_2. Mol. Wt. 76.14

A clear, colorless, neutral liquid of specific gravity 1.270 to 1.272, and boiling at 46° to 47° C. Carbon disulphide is insoluble in water, but is miscible in all proportions with alcohol and ether.

TESTS OF I URITY

Non-volatile Matter.* — On evaporating 50 c.c. of carbon disulphide on the water-bath, no weighable residue should remain. (Indicating less than 0.0008 per cent.)

Hydrogen Sulphide and Foreign Organic Sulphur Compounds. — (*a*) On shaking 10 c.c. of carbon disulphide with lead carbonate, the latter should not acquire a brown color. (Indicating none present.)

(*b*) On shaking 2 c.c. of carbon disulphide in a dry vessel with a globule of bright metallic mercury, the bright surface of the latter should not become covered with a dark, pulverulent coating. (Indicating none present.)

Sulphuric and Sulphurous Acids. — On shaking 10 c.c. of carbon disulphide with 5 c.c. of water, the latter should neither redden nor decolorize blue litmus paper. (Indicating none present.)

CARBON TETRACHLORIDE

CCl_4. Mol. Wt. 153.84.

A clear, colorless liquid only slightly soluble in water (about 0.08 parts to 100). Carbon tetrachloride is miscible in all proportions with absolute alcohol, ether, and with ethereal and fatty oils. Its specific gravity is 1.604; and its boiling-point, 76° to 77° C.

TESTS OF PURITY

Non-volatile Matter. — Upon evaporation on a water-bath 25 c.c. of carbon tetrachloride should leave no weighable residue. (Indicating less than 0.00125 per cent.)

Chlorine. — No blue color should appear upon shaking 20 c.c. of carbon tetrachloride with 5 c.c. of zinc iodide-starch solution. (Indicating less than 0.0002 per cent.)

Hydrochloric Acid. — After shaking 20 c.c. of carbon tetrachloride with 10 c.c. of water about one minute, the water should have a neutral reaction and should show no change upon addition of solution of silver nitrate. (Indicating less than 0.0001 per cent Cl.)

Organic Matter. — If 20 c.c. of carbon tetrachloride with 15 c.c. of concentrated sulphuric acid are frequently shaken in a glass-stoppered flask that has previously been rinsed with sulphuric acid, no color should appear in the acid within one hour. (Indicating none present.)

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* Under the influence of light, carbon disulphide becomes yellowish, and then leaves a slight residue on evaporation.

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Aldehyde. — If 10 c.c. of carbon tetrachloride and 10 c.c. of solution of potassium hydroxide (sp.gr. 1.3) are warmed for one minute with frequent shaking, neither a yellow nor a brown color should appear in the potassium hydroxide solution. (Indicating none present.)

Carbon Disulphide. — Allow a mixture of 10 c.c. of carbon tetrachloride and 10 c.c. of a solution of 10 gm. of potassium hydroxide in 100 gm. of absolute alcohol to stand about one hour. The subsequent addition to this of 5 c.c. of dilute acetic acid (sp.gr. 1.040–1.042) and some solution of copper sulphate should not develop a yellow precipitate within two hours. (Indicating less than 0.02 per cent CS_2.)

NOTE. — Regarding the quantitative determination of carbon disulphide in carbon tetrachloride see W. Schmitz-Dumont, Chem. Ztg., **21**, 487, 510 (1897); *abst.*, J. Chem. Soc., **74**, II, 140 (1898). L. G. Radcliffe, Z. angew. Chem., **22**, 1413 (1909); *abst.*, J. Chem. Soc., **96**, II, 438 (1909). J. Soc. Chem. Ind., **28**, 229 (1909); *abst.*, J. Chem. Soc., **96**, II, 438 (1909).

CARMINE

(Carmine 1. [Naccarat])

Bright red, light pieces, which are easily reduced to a fine powder. Carmine is insoluble in water, and in dilute acids, but is soluble in ammonia.

TESTS OF PURITY

Water. — 1 gm. of carmine should not lose more than 0.25 gm. in weight upon drying at 100° C. (Indicating not more than 25 per cent.)

Solubility. — 0.15 gm. should be almost completely soluble in a mixture of 5 c.c. of ammonia water and 20 c.c. of water, yielding a violet-red solution; only a small amount of flocculent matter should remain undissolved.

Ash. — 0.25 gm. of carmine cautiously incinerated in a porcelain crucible should not yield an ash weighing more than 0.020 gm. (Indicating not more than 8 per cent.) Particular attention should be paid to the odor of burning carmine; it is similar to that yielded during the combustion of proteids. An odor of bromine would point to a sophistication with an eosine-lake; and an odor of phenol would point to an admixture of pæonine-lake.

NOTE. — Regarding the adulterations of cochineal-carmine see E. Donath, Dingl. pol. J., **294**, 188 (1894); *abst.*, J. Soc. Chem. Ind., **14**, 305 (1895).

Regarding the composition of carmine ash see S. Feitler, Z. angew. Chem., **5**, 136 (1892) [or Z. anal. Chem., **32**, 627 (1893)]; *abst.*, J. Chem. Soc. Ind., **12**, 256 (1893).

Regarding the use of carmine as a staining reagent see Gould's Illustrated Dictionary of Medicine. 5 ed., 1389–1400 (1907).

CARMINE-FIBRIN

(Carmine-fibrin in Glycerol)

Dark, cherry-red masses, of a crumbling nature, and prepared by dyeing washed blood fibrin with an ammoniacal solution of carmine, as described by Grützner.

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Carmine-fibrin swells up, but does not dissolve or yield up its coloring matter, when treated with hydrochloric acid of 0.1 per cent strength. If pepsin, however, is added to this acid, the fibrin is more or less dissolved, depending on the quantity and strength of the pepsin and on the length of time allowed for solution to be effected. The progress of the reaction may be known from the color of the liquid, in which the carmine dissolves as rapidly as the fibrin is digested.

Carmine-fibrin should be washed with water before use, pressed out with the hands, and allowed to swell up completely in hydrochloric acid of 0.1–0.2 per cent strength. In the swollen condition it is cut up with shears.

NOTE. — Regarding the use of carmine-fibrin for the colorimetric valuation of pepsin see P. Grützner, Pflüger's Arch. ges. Physiol., 452 (1874). See also E. Merck's Annual Report, 46, on the year 1905. A. Korn, Inauguraldissertation, Tübingen (1902).

CHARCOAL, ANIMAL

(Blood Charcoal, Purified by Acid)

A dry, light, black powder.

TESTS OF PURITY

Material Soluble in Water. — Exhaust 1 gm. of animal charcoal by boiling with 20 c.c. of water for about five minutes under a reflux condenser. Filter and evaporate the filtrate to dryness. The residue should not weigh more than 0.003 gm. (Indicating not more than 0.3 per cent.)

Material Soluble in Alcohol. — Boil a mixture of 2 gm. of animal charcoal and 40 c.c. of alcohol (about 85 per cent by weight) for about five minutes under a reflux condenser; then filter. 20 c.c. of the filtrate should not leave more than 0.001 gm. of residue on evaporation. (Indicating not more than 0.1 per cent.)

Sulphates, Chlorides, and Nitrates. — Boil 1 gm. of animal charcoal with 50 c.c. of water for a few minutes, and filter. The filtrate should be colorless and neutral. On adding to 10 c.c. of the filtrate some barium nitrate solution, no immediate turbidity should be produced. (Indicating less than 0.05 per cent SO_3.)

On adding silver nitrate solution to 10 c.c. of the filtrate, not more than a slight opalescence should develop. (Indicating less than 0.01 per cent Cl.)

On adding a granule of sodium chloride, 1 drop of 1 : 1000 indigo solution and 10 c.c. of concentrated sulphuric acid to 10 c.c. of the filtrate, the blue color of the solution should not disappear upon shaking. (Indicating less than 0.016 per cent N_2O_5.)

Copper, Iron, and Calcium. — Boil 1 gm. of animal charcoal with 40 c.c. of water and 10 c.c. of hydrochloric acid for about five minutes, filter, and to 10 c.c. of the filtrate add 5 c.c. of ammonia water; the liquid should not acquire a blue color (indicating less than 0.002 per cent Cu), nor should a flocculent pre-

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cipitate form (indicating less than 0.02 per cent Fe). On the further addition of a few drops of ammonium oxalate solution no turbidity should develop immediately (indicating less than 0.025 per cent Ca.).

Residue on Ignition. — 1 gm. of animal charcoal should leave a residue weighing not more than 0.10 gm. on ignition. (Indicating not more than 10 per cent.)

Hydrogen Sulphide. — Heat 1 gm. of animal charcoal with 40 c.c. of water and 10 c.c. of hydrochloric acid, and test the escaping vapors with moistened lead acetate paper. The latter should not acquire a brown color. (Indicating less than 0.001 per cent S.)

Decolorizing Power. — Dissolve 0.2 gm. of Bismarck brown in 100 c.c. of water. Mix 0.1 gm. of animal charcoal with 40 c.c. of this, boil for 10 minutes under a reflux condenser, and then filter. The filtrate should be nearly colorless.

CHLORINE WATER

A clear, pale, greenish-yellow liquid, having a strong odor of chlorine. The liquid should contain about 0.4 per cent of Cl.

TESTS OF PURITY

Non-volatile Matter. — 20 gm. of chlorine water evaporated in a glass dish on a water-bath should leave no weighable residue. (Indicating less than 0.0025 per cent.)

Hydrochloric Acid.* — Shake 20 gm. of chlorine water with about 5 gm. of pure mercury vigorously for five minutes. Filter, add phenolphthalein to the filtrate, and run in normal potassium hydroxide solution, by drops, until a red color appears. Not more than 0.1 c.c. of normal alkali should be required to produce the red end-point. (Indicating less than 0.018 per cent HCl.)

Chlorine Content. — Let 25 gm. of chlorine water run into a flask containing a solution of 1 gm. of potassium iodide in 25 c.c. of water. Add a few drops of starch solution, and titrate the liberated iodine with decinormal sodium thiosulphate solution, of which at least 28.2 c.c. should be used to cause the blue color to disappear.

1 c.c. of decinormal $Na_2S_2O_3 = 0.003546$ gm. of Cl, log. 54974.

CHLOROFORM

$CHCl_3$. Mol. Wt. 119.39

A clear, colorless, very volatile liquid, slightly soluble in water (1 : 200), but miscible in all proportions with alcohol, ether, and fatty and volatile oils. Chloroform has a specific gravity of 1.485 to 1.489, and boils at 60° to 62° C.†

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* Chlorine water, unless recently prepared, always contains hydrochloric acid.

† The readiness of pure (absolute) chloroform to decompose may be prevented by the addition of a small amount (up to 1 per cent) of pure absolute alcohol. Chloroform of the above specific gravity contains about 1 per cent of alcohol. The alcohol may be removed by shaking it with water, then with pure sulphuric acid, then separating the chloroform and mixing it with granulated potassium carbonate, and distilling.

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TESTS OF PURITY

Non-volatile Matter. — 25 c.c. of chloroform upon evaporation on a water-bath should leave no weighable residue. (Indicating less than 0.0014 per cent.)

Hydrochloric Acid. — Vigorously shake together 20 c.c. of chloroform and 10 c.c. of water for about a minute; after the chloroform has separated, draw off the aqueous layer with a pipette. The aqueous liquid should not redden blue litmus paper, nor should it be changed on the addition of silver nitrate solution. (Indicating less than 0.0001 per cent HCl.)

Free Chlorine. — On shaking 20 c.c. of chloroform with 5 c.c. of zinc iodide-starch solution, the latter should not acquire a blue color. (Indicating less than 0.0002 per cent Cl.)

Phosgen. — If 10 c.c. of chloroform are overlaid with clear baryta water, no white film should form at the contact-surfaces of the two liquids. (Indicating none present.)

Aldehyde. — 10 c.c. of chloroform and 10 c.c. of solution of potassium hydroxide (sp.gr. 1.3) are warmed for one minute while vigorously stirred. Neither a brown nor yellow color should appear in the potassium hydroxide solution. (Indicating none present.)

Foreign Organic Bodies. — On repeatedly shaking 20 c.c. of chloroform, 15 c.c. of concentrated sulphuric acid, and 4 drops of formaldehyde, in a glass-stoppered flask, which has been rinsed previously with sulphuric acid, the acid should not acquire a color within one-half hour. (Indicating none present.)

CHROMIUM TRIOXIDE FOR CARBON DETERMINATION

(Acid Chromic for Carbon Determination)

CrO_3. Mol. Wt. 100.1

Dark, brownish-red, hygroscopic needles or rhombic prisms, which are very easily soluble in water. The preparation contains at least 80 per cent of CrO_3.

TESTS OF PURITY

Carbon. — This test is to be carried out as described by Lunge, [Chem.-tech. Untersuch.-Meth., 5 ed., 2, 56 (1904)] in his Estimation of Carbon in Iron by Combustion in the Wet Way. For the test use a solution of 15 gm. of chromium trioxide in 150 c.c. of water and 200 c.c. of concentrated sulphuric acid (sp.gr. 1.84). The use of a wash bottle of concentrated sulphuric acid between the combustion tube and the U-tube of glacial phosphoric acid is recommended.

Quantitative Determination. — This test is to be made in the manner described under Chromium Trioxide, Free from Sulphuric Acid.

CHROMIUM TRIOXIDE, FREE FROM SULPHURIC ACID

([Acid] Chromic Anhydride)

CrO_3. Mol. Wt. 100

Dark, brownish-red, hygroscopic needles, or rhombic prisms, very readily soluble in water. Chromium trioxide contains almost 100 per cent of CrO_3.

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TESTS OF PURITY

Sulphuric Acid. — 10 gm. of chromium trioxide must afford a clear solution with 100 c.c. of water. On adding to the solution 20 c.c. of hydrochloric acid and some barium chloride solution, no turbidity should occur within fifteen hours. (Indicating less than 0.005 per cent SO_3.)

Potassium Sulphate and Potassium Chromate. — Ignite 0.2 gm. of chromium trioxide in a porcelain crucible, triturate the residue with about 20 c.c. of water, and filter. Evaporate the filtrate to dryness on a water-bath, dry the residue at 100° C., and weigh. The residue must not weigh more than 0.002 gm. (Indicating not more than 1 per cent.)

Quantitative Determination. — Dissolve 1 gm. of chromium trioxide in water and dilute to 100 c.c. Introduce 10 c.c. of this solution into a stoppered flask of about 400 c.c. capacity, dilute with 100 c.c. of water, and add 5 gm. of potassium iodide and 2 c.c. of hydrochloric acid. Allow the mixture to stand for about ten minutes with frequent shaking, then dilute with 200 c.c. of water, and titrate with decinormal sodium thiosulphate solution.

1 c.c. of decinormal $Na_2S_2O_3 = 0.003333$ gm. of CrO_3, log. 52288.

COBALT NITRATE

(Cobaltous Nitrate)

$Co(NO_3)_2 + 6H_2O$. **Mol. Wt. 291.09**

Red, monoclinic prisms, deliquescent in moist air, and readily soluble in water and alcohol.

I

COBALT NITRATE

TESTS OF PURITY

Sulphates. — A solution of 1 gm. of cobalt nitrate in 20 c.c. of water, to which have been added 1 c.c. of hydrochloric acid and a little barium chloride solution, should not become turbid. (Indicating less than 0.01 per cent SO_3.)

Chlorides. — The 1 : 20 aqueous solution, acidulated with nitric acid, should show at most a faint opalescence upon addition of silver nitrate solution. (Indicating less than 0.002 per cent Cl.)

Alkali Salts. — Precipitate the cobalt completely from a solution of 2 gm. of cobalt nitrate in 100 c.c. of water by adding ammonia water and ammonium sulphide solution; filter, evaporate the filtrate to dryness, and ignite the residue. The weight of the latter should not exceed 0.005 gm. (Indicating not more than 0.25 per cent.)

Zinc. — To a solution of 0.5 gm. of cobalt nitrate in 50 c.c. of water, add 5 c.c. of sodium hydroxide solution (sp.gr. 1.3); filter, and to the filtrate add ammonium sulphide solution. No precipitate should form. (Indicating less than 0.5 per cent Zn.)

Lead and Copper. — Dissolve 2 gm. of cobalt nitrate in 50 c.c. of water, and add 2 c.c. of nitric acid, followed by hydrogen sulphide water. The solution must show no change. (Indicating less than 0.02 per cent Pb, or less than 0.002 per cent Cu.)

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II

COBALT NITRATE, FREE FROM NICKEL

TESTS OF PURITY

Nickel. — To a solution of 1 gm. of cobalt nitrate in 20 c.c. of water add 1.5 gm. of potassium cyanide and heat until a yellow color develops. Filter, cool, add 1 c.c. of bromine, and shake until the bromine is dissolved. The subsequent addition of 10 c.c. of solution of sodium hydroxide (sp.gr. 1.168 to 1.172) should not cause a black precipitate. (Indicating less than 0.02 per cent Ni.)

The tests for sulphates, chlorides, alkali salts, zinc, lead, and copper are also to be made, as described and conditioned under Cobalt Nitrate, page 71.

COBALT OXIDE

Co_2O_3. Mol. Wt. 165.94

A fine, black powder free from sulphur. It is used to determine sulphur in coal and coke according to the method of Brunck.*

TEST OF PURITY

Sulphur. — Mix 5 gm. of cobalt oxide and 2.5 gm. of anhydrous sodium carbonate in a mortar and then ignite the mixture in a platinum crucible.† The melt is leached with about 50 c.c. of hot water, the solution filtered, and, after acidulation with hydrochloric acid, is treated with solution of barium chloride. No precipitate of barium sulphate should be visible after standing fifteen hours. (Indicating less than 0.0006 per cent S.)

COLLODION

A colorless, or slightly yellowish, neutral syrupy liquid, which, exposed in thin layers, evaporates, and leaves a tough, colorless film. Collodion contains about 4 per cent of soluble cotton.

TESTS OF PURITY

Acids. — Blue litmus paper, when immersed in collodion, should not be reddened immediately. (Indicating none present.)

Determination of Residue. — Mix 10 c.c. of water by drops and with constant stirring with 10 gm. of collodion and then evaporate to dryness on the water-bath. The residue when dried at 95° to 100° C. should weigh 0.38 to 0.40 gm. (Indicating 3.8 to 4.0 per cent.)

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* O. Brunck, Z. angew. Chem., 18, 1560 (1905); *abst.*, J. Chem. Soc., 88, II, 762 (1905).

† In order to prevent the alkaline contents of the crucible from taking up sulphur from the flame gases, the crucible is allowed to rest during ignition in a circular opening in an asbestos plate.

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COPPER, BY ELECTROLYSIS

Cu. Atomic Wt. 63.57

TESTS OF PURITY

Foreign Metals.* — (*a*) Dissolve 10 gm. of copper in 60 c.c. of nitric acid (sp.gr. 1.3), and evaporate the solution to dryness on the water-bath. The residue should completely dissolve in 50 c.c. of water and 10 c.c. of nitric acid (sp.gr. 1.3), yielding a clear solution (indicating no antimony and tin present). To the solution add 15 c.c. of concentrated sulphuric acid (sp.gr. 1.84), evaporate on the water-bath as far as possible, heat the residue on a sand-bath until vapors of sulphuric acid begin to be evolved, and take up the residue with 100 c.c. of water. No insoluble residue should remain (indicating no lead present). On adding 5 c.c. of hydrochloric acid to the clear solution, no turbidity should ensue (indicating no silver present). Now add 150 c.c. of ammonia water to the liquid, allow to stand three to four hours at 50 to 60° C., filter through an ashless filter, and wash with ammoniacal water until perfectly free from copper. A precipitate on the paper at this point is removed from the paper by dissolving in a little hydrochloric acid, and the paper is washed with water. The mixed acid and washings are made ammoniacal again and filtered through the same paper. Incinerate the filter with any precipitate it may contain, and ignite. The residue should not weigh more than 0.001 gm. (Indicating not more than 0.01 per cent iron and bismuth as Fe_2O_3 and Bi_2O_3.)

(*b*) Dissolve 10 gm. of copper in 60 c.c. of nitric acid (sp.gr. 1.3), add to the solution 15 c.c. of concentrated sulphuric acid (sp.gr. 1.84), evaporate the solution on the water-bath as far as possible, and heat the residue on a sand-bath until the vapors of sulphuric acid begin to be evolved. Dissolve the residue in 300 c.c. of water, pass hydrogen sulphide gas into the solution at 70° C. until the copper is completely precipitated, filter, concentrate the filtrate, expel the sulphuric acid on the sand-bath, and ignite the residue. The weight of the latter should not exceed 0.002 gm. (Indicating not more than 0.02 per cent.)

Arsenic. — Dissolve 10 gm. of copper in 60 c.c. of nitric acid (sp.gr. 1.3), add to the solution 15 c.c. of concentrated sulphuric acid (sp.gr. 1.84), evaporate the solution on the water-bath as far as possible, and then heat the residue on the sand-bath until vapors of sulphuric acid are evolved. When cold, dissolve the residue in 100 c.c. of water.

Set a Marsh apparatus in operation, using 50 gm. of arsenic-free, granulated zinc, and dilute (1 : 5) sulphuric acid, then introduce the copper sulphate solution in small quantities at a time, and maintain the flow of gas for one hour. No deposit of arsenic should be visible in the reduction tube within this time. (Indicating less than 0.0001 per cent As.)

COPPER CHLORIDE, CUPRIC

(Copper Dichloride)

$CuCl_2+2H_2O$. Mol. Wt. 170.52

Green, hygroscopic crystals, easily soluble in water and in alcohol.

TESTS OF PURITY

Substances Insoluble in Alcohol. — The solution of 5 gm. of copper chloride

* Wash the copper in diluted hydrochloric acid before using it in this test.

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in 5 c.c. of water should not be rendered turbid on mixing with 5 c.c. of alcohol (about 85 per cent by weight). (Indicating none present.)

Sulphates. — Dissolve 1 gm. of copper chloride in 20 c.c. of water, and add 1 c.c. of hydrochloric acid, followed by barium chloride solution. No turbidity should ensue within five minutes. (Indicating less than 0.01 per cent SO_3.)

Salts of Alkali Metals. — Dissolve 3 gm. of copper chloride in 100 c.c. of water, add 5 c.c. of hydrochloric acid, and into the solution, maintained at about 70° C., pass hydrogen sulphide gas, until the copper is completely precipitated; filter, evaporate the filtrate to dryness, and ignite the residue. The weight of the latter should not exceed 0.002 gm. (Indicating not more than 0.066 per cent.)

Arsenic. — Introduce 20 gm. of arsenic-free, granulated zinc into the generating flask of a Marsh apparatus, and start the hydrogen by adding dilute (1 : 5) sulphuric acid. Dissolve 1 gm. of copper chloride in 20 c.c. of water, introduce the solution in small quantities at a time into the Marsh apparatus, and maintain a slow stream of gas for one hour. No deposit of arsenic should be visible in the reduction tube of the apparatus within this time. (Indicating less than 0.001 per cent As.)

Iron. — Heat to boiling a solution of 5 gm. of copper chloride in 25 c.c. of water containing 2 c.c. of nitric acid (sp. gr. 1.3), then add 35 c.c. of ammonia water. Filter, and wash with ammoniacal water until the copper is all removed from the paper. Any precipitate remaining on the paper is dissolved in a little hydrochloric acid, the paper washed, and the acid and washings after mixing are treated with an excess of ammonia water. Any precipitate is filtered through the original paper. The paper and precipitate are ignited, leaving a residue weighing not more than 0.002 gm. (Indicating not more than 0.028 per cent Fe.)

COPPER CHLORIDE, CUPROUS

(Copper Monochloride)

Cu_2Cl_2. Mol. Wt. 198.06

A white, crystalline powder, insoluble in water, but soluble in concentrated hydrochloric acid and in ammonia water.

The quality of cuprous chloride may be judged from its appearance. The preparation should be white, but not green or brown.* Both the hydrochloric acid and the ammoniacal solutions should rapidly and freely absorb carbon monoxide.

COPPER HYDROXIDE

Light blue paste prepared according to Fassbender's method.†

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* Cuprous chloride soon acquires a green color in air.

† G. Fassbender, Ber., **13**, 1821 (1880); *abst.*, J. Chem. Soc., **40**, 205 (1881). See also A. Stutzer, Ber., **13**, 251 (1880); *abst.*, J. Chem. Soc., **38**, 676 (1880); Chem. Ztg., **4**, 360 (1880) [or Z. anal. Chem., **20**, 307 (1880)]; *abst.*, J. Chem. Soc., **40**, 660 (1881). E. Schulze and J. Barbieri, Landw. Vers. Sta., **26**, 213–283 [or Z. Anal. Chem., **20**, 588 (1881)]; *abst.*, J. Chem. Soc., **40**, 312 (1881).

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TEST OF PURITY

Sulphates, Alkalies. — Shake 50 c.c. of water with 10 gm. of the copper hydroxide and then filter. The filtrate should be neutral (indicating no free alkalies present) and show no change upon the addition of solution of barium chloride. (Indicating less than 0.001 per cent SO_3.)

COPPER OXIDE

(Cupric Oxide)

CuO. Mol. Wt. 79.57

Copper oxide is used in ultimate organic analysis, in the form of fine powder or coarse granules, and also in the form of wire.

I

COPPER OXIDE, POWDER

A black powder.

TESTS OF PURITY

Nitrates and Carbon Dioxide. — On heating 100 gm. of copper oxide in a glass tube, and passing over it a stream of moist air freed from carbonic acid, no vapors should be given off which redden litmus paper, or render lime-water turbid. (Indicating none present.)

Chlorides. — The solution of 1 gm. of copper oxide in 10 c.c. of nitric acid and 10 c.c. of water should not be rendered cloudy by silver nitrate solution. (Indicating less than 0.0005 per cent Cl.)

Substances not Precipitated by Hydrogen Sulphide (Iron, Insoluble Matter, etc.). — Dissolve 2 gm. of copper oxide in 10 c.c. of hydrochloric acid (sp.gr. 1.19), dilute with water to 100 c.c.; ignite the insoluble residue, the weight of which should not exceed 0.001 gm. (Indicating not more than 0.05 per cent insoluble matter.) Pass hydrogen sulphide gas into the solution at about 70° C., until the copper has completely precipitated, filter, evaporate the filtrate on the water-bath, and ignite the residue. The weight of the latter should not exceed 0.005 gm. (Indicating not more than 0.25 per cent.)

Soluble Substances (Alkali Salts, etc.). — Shake 3 gm. of copper oxide with 60 c.c. of water a few minutes and then filter. The filtrate should not react alkaline with litmus, and upon evaporation and ignition should leave no weighable residue. (Indicating less than 0.0167 per cent.)

Sulphates. — Dissolve 1 gm. of copper oxide in 5 c.c. of hydrochloric acid (sp.gr. 1.19), dilute with 20 c.c. water and add barium chloride solution. No change should appear. (Indicating less than 0.08 per cent SO_3.)

Calcium. — Digest 20 gm. of copper oxide with a mixture of 5 c.c. of nitric acid and 95 c.c. of water for about fifteen minutes, shaking frequently, filter, precipitate the copper in the filtrate completely by passing hydrogen sulphide gas, and filter again. Evaporate the filtrate on the water-bath to about 20 c.c., add ammonia water in excess, filter once more, and to the filtrate add ammonium oxalate solution. No immediate turbidity should be produced. (Indicating less than 0.00125 per cent Ca.)

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II

COPPER OXIDE, GRANULAR

Grayish-black granules.

TESTS OF PURITY

The tests for nitrates, carbon dioxide, and calcium are to be made as described under copper oxide, powder. The granules are to be powdered before testing.

Sulphates. — Dissolve 1 gm. of granulated copper oxide in 5 c.c. of hydrochloric acid (sp.gr. 1.19), dilute with 20 c.c. of water and filter. The filtrate should become at most slightly opalescent upon the addition of barium chloride solution. (Indicating less than 0.1 per cent SO_3.)

Chlorides. — 1 gm. of granulated copper oxide is powdered and shaken a few minutes with 10 c.c. of nitric acid and 10 c.c. of water. After filtering, silver nitrate solution should not produce a cloud in the filtrate. (Indicating less than 0.005 per cent Cl.)

III

COPPER OXIDE, WIRE

This occurs in the form of short cylindrical pieces.

COPPER SULPHATE

(Cupric Sulphate)

$CuSO_4+5H_2O$. Mol. Wt. 249.72

Blue, transparent crystals, slightly efflorescent in dry air, soluble in 3.5 parts of cold, and in 1 part of boiling, water; almost insoluble in alcohol. The aqueous solution is acid to litmus paper.

TESTS OF PURITY

Salts of the Alkalies, Earths, etc. — Dissolve 3 gm. of copper sulphate in 100 c.c. of water, add 5 c.c. of hydrochloric acid, and into the solution, maintained at about 70° C., pass hydrogen sulphide gas until the copper is completely precipitated. On evaporating the filtrate to dryness, and igniting the residue, the weight of the latter should not exceed 0.001 gm. (Indicating not more than 0.033 per cent.)

Iron. — To the solution of 5 gm. of copper sulphate in 25 c.c. of water, add 2 c.c. of nitric acid (sp.gr. 1.3), and heat the mixture to boiling. Then add 20 c.c. of ammonia water, pour the liquid through an ashless filter, and wash the latter with ammoniacal water until perfectly free from copper. Any precipitate on the filter paper is dissolved in a little hydrochloric acid, the paper washed with water, and the mixed acid and washings are treated again with excess of ammonia water. Filter through the same paper. Incinerate and ignite the filter together with any precipitate it may contain. The weight of the residue should not exceed 0.001 gm. (Indicating not more than 0.014 per cent Fe.)

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COPPER AND AMMONIUM CHLORIDE

(Ammonio-Cupric Chloride)

$CuCl_2 2NH_4Cl + 2H_2O$. Mol. Wt. 277.51

Blue crystals soluble in 4 parts of water, yielding a solution slightly acid to litmus paper.

TESTS OF PURITY

Free Acids. — A solution of 30 gm. of copper and ammonium chloride in 100 c.c. of water must be perfectly clear. On introducing into the solution several pieces of piano wire, the latter should dissolve with the deposition of copper, but without any evolution of gas. (Indicating none present.)

Sulphates. — Dissolve 1 gm. of copper and ammonium chloride in 20 c.c. of water, and add 1 c.c. of hydrochloric acid, followed by barium chloride solution. No turbidity should be produced within five minutes. (Indicating less than 0.01 per cent SO_3.)

Salts of the Alkalies, Earths, etc. — Dissolve 3 gm. of copper and ammonium chloride in about 100 c.c. of water, add 5 c.c. of hydrochloric acid, pass hydrogen sulphide gas into the solution at about 70° C., until the copper is completely precipitated; filter, evaporate the filtrate, and ignite the residue. The weight of the latter should not exceed 0.002 gm. (Indicating not more than 0.067 per cent.)

Iron. — Dissolve 5 gm. of copper and ammonium chloride in 25 c.c. of water, add 2 c.c. of nitric acid (sp.gr. 1.3), heat to boiling, then add 20 c.c. of ammonia water and pour the liquid through an ashless filter. Wash the latter with ammoniacal water until perfectly free from copper, dissolve any precipitate on the paper with hydrochloric acid, wash the paper with water, and then add an excess of ammonia water to the mixed hydrochloric acid and washings. Filter through the same paper, and then incinerate and ignite the filter together with any precipitate it may contain. The weight of the residue should not exceed 0.001 gm. (Indicating not more than 0.014 per cent Fe.)

DEVARDA'S METAL

(Devarda's Alloy)

Devarda's metal is a gray powder composed of 50 parts of copper, 45 parts of aluminum, and 5 parts of zinc. The powder is partly soluble in hydrochloric acid, copper separating out.

TEST OF PURITY

Nitrogen. — Place 10 gm. of Devarda's metal in an Erlenmeyer flask of about 1 liter capacity, add 100 c.c. of hydrochloric acid, and allow to stand with frequent agitation until hydrogen ceases to be evolved. After cooling add 100 c.c. of nitrogen-free solution of sodium hydroxide (sp.gr. 1.3) and distil about 50 c.c., receiving the distillate in 2 to 3 c.c. of fifth-normal solution of hydrochloric acid diluted with 10 c.c. of water. Titrate the distillate with fifth-normal solution of potassium hydroxide, using methyl orange as indicator. The ammonia from

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the metal should not consume more than 0.2 c.c. of fifth-normal solution of hydrochloric acid. (Indicating not more than 0.0056 per cent N.)

NOTE. — Regarding the use of Devarda's metal for the estimation of nitrogen see A. Devarda, Chem. Ztg., **16**, 1952 (1892); *abst.*, J. Chem. Soc., **64**, 343 (1893). Österr.-Ungar. Z. Zucker-Ind., 1897; *abst.*, J. Chem. Soc., **74**, II, 350 (1898). Meissl, Landwirtsch. Versuchsta., **42**, 97 (1893); *abst.*, Z. anal. Chem., **33**, 113 (1894). Compare G. Lunge, Chem.-Tech. Untersuch. Meth., 6 ed, **3**, 11 (1911).

DICYANDIAMIDINE SULPHATE

$(C_2OH_4N_4)_2H_2SO_4+2H_2O$. Mol. Wt. 338.29

A white powder, soluble in about 20 parts of cold, and in about 3 parts of boiling, water.

TEST OF PURITY

Suitability as a Reagent for Nickel.—Mix 10 c.c. of a solution of cobalt-free nickel chloride (2.4 gm. of $NiCl_2+6H_2O$ in 100 c.c. of water) with 3 c.c. of solution of ammonium chloride, 10 c.c. of 20 per cent ammonia water, and 10 c.c. of solution of dicyandiamidine sulphate (4.5 gm. in 125 c.c. of water). Add to this mixture, while constantly stirring, 6 c.c. of solution of potassium hydroxide (sp.gr. 1.138–1.140), allow to stand about fifteen hours, and then filter. The filtrate, to which 10 c.c. of dicyandiamidine sulphate solution have been added, and which has then been allowed to stand six hours, should not exhibit a yellow precipitate.

NOTE. — Regarding the use of dicyandiamidine sulphate for the detection and quantitative determination of nickel, as well as for its separation from cobalt and other metals, see H. Grossman and B. Schück, Ber., **39**, 3356 (1906); *abst.*, J. Chem. Soc., **90**, II, 903 (1906). Chem.-Ztg., **31**, 535, 911 (1907); *abst.*, J. Chem. Soc., **92**, II, 819 (1907). H. Grossman and W. Heilborn, Ber., **41**, 1878 (1908); *abst.*, J. Chem. Soc., **94**, II, 635 (1908). Chem.-Ztg., **33**, 841, 851 (1909); *abst.*, C. A., **3**, 2782 (1909).

DIMETHYLGLYOXIME

(Diacetyl-dioxime)

$CH_3C(NOH)C(NOH)CH_3$. Mol. Wt. 116.08

White, needle-shaped crystals almost insoluble in cold water and very difficultly soluble in boiling water. Difficultly soluble also in alcohol and in ether. Dimethylglyoxime melts at 241° C., but with a brown discoloration.

TEST OF PURITY

Suitability as a Reagent for Nickel.—Dissolve 2.4 gm. of cobalt-free nickel chloride ($NiCl_2+6H_2O$) in 100 c.c. of water, draw off 10 c.c. of this solution and, after diluting with 100 c.c. of water, heat to boiling and add a solution of 0.25 gm. of dimethylglyoxime in 25 c.c. of alcohol (about 85 per cent by weight). Add ammonia water by drops until the solution is alkaline, and, after cooling,

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filter. The filtrate, when heated to boiling with alcoholic solution of dimethylglyoxime (1 : 100), should show no brown color, and, upon subsequent cooling, should exhibit no red precipitate.

NOTE. — Regarding the use of dimethylglyoxime for the detection and quantitative determination of nickel see O. Brunck, Z. angew. Chem., **20**, 834 (1907); *abst.*, J. Chem. Soc., **92**, II, 582 (1907). Z. angew. Chem., **20**, 1844 (1907); *abst.*, J. Chem. Soc., **92**, II, 989 (1907). P. Bogolubow, Stahl u. Eisen, **30**, 458 (1910); *abst.*, Chem. Zentr. (5) 14, 1643 (1910); *abst.*, C. A., **5**, 1572 (1911). L. Tschugaeff, Ber., **28**, 2520 (1905); *abst.*, J. Chem. Soc., 88, II, 613 (1905). K. Kraut, Z. angew. Chem., **19**, 1793 (1906); *abst.*, J. Chem. Soc., **90**, II, 858 (1906).

DIMETHYLPARAPHENYLENEDIAMINE HYDROCHLORIDE

(Para-aminodimethylaniline Hydrochloride)

$C_6H_4(NH_2)N(CH_3)_2+2HCl$. Mol. Wt. 209.05

A white, or grayish, hygroscopic, crystalline powder, easily soluble in water, but less soluble in alcohol. The article must be protected from air and moisture.

TESTS OF PURITY

Non-volatile Matter. — 1 gm. of dimethylparaphenylenediamine hydrochloride upon ignition should not leave a weighable residue. (Indicating less than 0.05 per cent.)

Quantitative Determination. — Titrate the solution of 0.5 gm. of dimethylparaphenylenediamine hydrochloride in 50 c.c. of alcohol (about 85 per cent by weight) in a glass-stoppered flask, using fifth-normal solution of potassium hydroxide, and Poirrier's blue as indicator.

1 c.c. of fifth-normal potassium hydroxide solution = 0.020905 gm. of $C_6H_4(NH_2)(CH_3) + HCl$, log. 32025.

NOTE. — Regarding the use of dimethylparaphenylenediamine hydrochloride as a reagent see A. Jolles, Detection of Bromine in Urine, Z. anal. Chem., **37**, 439 (1898); *abst.*, J. Chem. Soc., **74**, II, 637 (1898). H. N. Malerba, Detection of Acetone, Ann. Chim. Farm., **21**, 14 (1895); *abst.*, Chem. Ztg., **19**, Repert., 82 (1895); *abst.*, J. Soc. Chem. Ind., **14**, 599 (1895). M. Pabst, Reagent for Olive Pits in Powdered Pepper, Chem. Zentr., (4) **11**, 1074 (1890); *abst.*, J. Soc. Chem. Ind., **9**, 770 (1890). C. Wurster, Quantitative Estimation of Wood in Paper, Ber., **20**, 808 (1887); *abst.*, J. Chem. Soc., **52**, 620 (1887). C. Wurster, Reagent for Detecting Minimal Quantities of Active Oxygen, Ber., **19**, 3195 (1887) and **21**, 921 (1888); *abst.*, J. Chem. Soc., **52**, 295 (1887) and **54**, 627 (1888).

DIPHENYLAMINE*

$(C_6H_5)_2NH$. Mol. Wt. 169.10

White, monoclinic crystals, insoluble in water, but easily soluble

* The diphenylamine solution, used as a reagent for nitric acid, is prepared by dissolving 0.5 gm. of diphenylamine in 100 c.c. of pure, concentrated sulphuric acid (free from nitric acid) and 20 c.c. of water. According to G. Lunge [Z.

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in alcohol, ether, and benzene. Diphenylamine melts at 53° C., and boils at 302° C.

TESTS OF PURITY

Nitric Acid. — 0.2 gm. of diphenylamine should dissolve in 20 c.c. of pure concentrated sulphuric acid with 2 c.c. of water, and yield a colorless solution. (Indicating less than 0.05 per cent N_2O_5.) Should a blue color develop, the sulphuric acid should be tested with brucine for the possible presence of nitric acid.

Aniline. — On pouring 1 gm. of powdered diphenylamine into 20 c.c. of a chlorinated lime solution, the liquid should not acquire a violet color. (Indicating less than 0.05 per cent.)

ETHER

(Ethyl Ether)

$(C_2H_5)_2O$. Mol. Wt. 74.08

I

ETHER, Sp.Gr. 0.720

A clear, colorless, mobile liquid of specific gravity 0.720,* boiling at 34° to 36° C. Ether should not redden moist blue litmus paper. Filter paper which has been moistened with ether should have no odor when dry.

TESTS OF PURITY

Residue. — On allowing 20 c.c. of ether to evaporate spontaneously in a glass dish covered with a funnel, the residue must have no odor, and should not redden or decolorize blue litmus paper; and it must be completely volatilized on warming on the water-bath. (Indicating none present.)

Ethyl Peroxide, Hydrogen Peroxide, and Ozone. — On vigorously shaking 10 c.c. of ether with 1 c.c. of 1 : 10 aqueous potassium iodide solution in a completely filled glass-stoppered bottle, neither the ether nor the potassium iodide solution should acquire a color after standing one hour in the dark. (Indicating none present.)

Aldehydes and Vinyl Alcohol. — Upon shaking 20 c.c. of ether and 5 c.c. of Nessler's solution one minute in a glass-stoppered flask, neither a reddish-brown nor a black precipitate should appear. (Indicating none present.)

Sulphur Compounds. — On shaking 20 c.c. of ether with a globule of mercury

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angew. Chem., **7**, 345 (1894); *abst.*, J. Chem. Soc., **66**, II, 398 (1894)], the reagent is employed as follows:

To test for nitric or nitrous acid, introduce about 5 c.c. of the reagent solution into a test-tube, and overlay it with the liquid to be examined. If the latter is specifically heavier than the reagent solution, the diphenylamine solution is to be overlaid upon the liquid.

Regarding the increased sensitiveness of the nitric-acid reaction with diphenylamine in water analysis, see R. Cimmino, Z. anal. Chem. **38**, 429 (1899); *abst.*, J. Chem. Soc., **70**, II, 805 (1899).

*Ether eagerly abstracts moisture from the atmosphere, whereby its specific gravity is increased.

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for two minutes in a glass-stoppered bottle, the bright surface of the metal must not be changed; nor should a black precipitate form. (Indicating none present.)

Acetone. — Mix 6 c.c. of baryta water and 6 drops of solution of mercuric chloride (1 : 20) with 20 c.c. of ether for one minute. Allow the liquids to separate, filter the aqueous portion, and to the clear filtrate add solution of ammonium sulphide. No dark color should appear within ten minutes. (Indicating less than 0.005 per cent.)

Water. — On shaking 20 c.c. of ether in a stoppered flask with 1 gm. of anhydrous copper sulphate, the latter should not acquire a green or blue color. (Indicating less than 1%.)

II

ETHER, ANHYDROUS, DISTILLED OVER SODIUM

TESTS OF PURITY

In addition to the tests given above, this preparation must answer the following requirement:

Water and Alcohol.—On introducing 15 c.c. of ether into a dry, glass-stoppered flask with a freshly-cut piece of sodium the size of a pea, only a slight evolution of gas should take place, and the bright metallic surfaces of the sodium should not completely lose their luster within six hours. (Indicating less than 0.1 per cent water and less than 0.3 per cent alcohol.)

FURFURAL

(Furfurol)

$C_5H_4O_2$. Mol. Wt. 96.03

When freshly prepared, furfural is a clear, colorless liquid; it rapidly acquires a yellow color on exposure to light and air. Furfural has a specific gravity of 1.165 to 1.166, boils at 158° to 160° C., and is soluble in 15 parts of cold water. It is very easily soluble in alcohol and in ether.

NOTE. — Regarding the quantitative determination of furfural, see W. E. Stone, Z. anal. Chem., **40**, 550 (1901); *abst.*, J. Anal. Appl. Chem., **5**, 421 (1895). W. Cormack, Z. anal. Chem., **43**, 256 (1904); *abst.*, J. Chem. Soc., **77**, 990 (1900). Regarding the use of furfural: (1) As a reagent for sesame oil, see F. Utz, Pharm. Ztg., **45**, 490 (1900); *abst.*, J. Chem. Soc., **78**, II, 699 (1900); also see Lewkowitsch, Chem. Technol. and Anal. Oils, Fats, and Waxes, 3 ed., Vol. II, p. 542 (1904). (2) As a reagent for alkaloids, see N. Wender, Chem. Ztg., **17**, 950 (1893); *abst.*, J. Chem. Soc., **66**, II, 76 (1894); Woltering, Pharm. Ztschr. f. Russl., **31**, or 526 (Z. anal. Chem., **36**, 410 (1897). (3) As a reagent for urea, see H. Schiff, J. Chem. Soc., **52**, 571 (1887). (4) As a reagent for fusel oil in alcohol, see A. Komarowsky, Chem. Ztg., **27**, 808 (1903); *abst.*, J. Chem. Soc., **84**, II, 700 (1903). (5) As a reagent for mineral acids, see O. Carletti, Boll. Chim. Farm., **45**, 449; *abst.*, C. A., I., 1458 (1907). E. Eggers, Z. anal. Chem., **27**, 725 (1888); *abst.*, J. Chem. Soc., **56**, 648 (1889).

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GALLEIN DRY

(Gallein)

Gallein is a brown powder soluble in alcohol. It is used as an indicator in the form of a solution of 0.5 gm. of gallein dissolved in a mixture of 50 c.c. of alcohol (about 85 per cent by weight) and 50 c.c. of water.

NOTE. — Regarding the use of gallein as an indicator, see H. Dechan, Pharm. J. Trans., (3), **15**, 849 (1885); *abst.*, Chem. Ztg., **9**, 715 (1885); *abst.*, J. Chem. Soc., **48**, 1012 (1885). Compare Böckmann, Chem.-tech. Untersuch. Meth., 3 ed., I, 142.

GLASS WOOL

Glass wool consists of fine glass threads.

TESTS OF PURITY

Matter Soluble in Hydrochloric Acid. — Boil 5 gm. of glass wool a few minutes with a mixture of 100 c.c. of hydrochloric acid (sp.gr. 1.124–1.126) and 100 c.c. of water. Filter, and evaporate 100 c.c. of the filtrate to dryness. The residue should not weigh more than 0.02 gm. (Indicating not more than 0.8 per cent.)

Lead. — Boil 1 gm. of glass wool a few minutes with a mixture of 10 c.c. of nitric acid (sp.gr. 1.150–1.152) and 40 c.c. of water. Filter, evaporate the filtrate to dryness on the water-bath, and dissolve the residue in 10 c.c. of water. After filtering again the solution should show no change upon the addition of hydrogen sulphide water. (Indicating less than 0.0025 per cent Pb.)

GLYCERIN

(Glycerol)

$C_3H_8O_3$. Mol. Wt. 92.06

I

GLYCERIN, Sp.Gr. 1.250

A clear, colorless, almost odorless liquid, neutral in reaction, soluble in all proportions in water and in alcohol. Insoluble in ether and in chloroform. Specific gravity not less than 1.250. It contains at least 95 per cent of absolute glycerin, $C_3H_8O_3$.

TESTS OF PURITY

Arsenic. — On mixing 1 c.c. of glycerin with 3 c.c. of stannous chloride solution the mixture should not darken within one hour. (Indicating less than 0.0012 per cent As.)

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Neutrality. — On diluting 2 c.c. of glycerin with 10 c.c. of water, the solution should not affect red or blue litmus paper.

Inorganic Matter. — On heating 5 c.c. of glycerin to boiling in an open dish and then igniting the vapors, the glycerin should burn up completely, leaving only a dark stain which should disappear on being more strongly heated. No weighable residue should remain. (Indicating less than 0.008 per cent.)

Substances which Reduce Ammoniacal Silver Nitrate Solution. — On heating a mixture of 1 c.c. of glycerin and 1 c.c. of ammonia water on the water-bath to 60° C., and then immediately adding 3 drops of silver nitrate solution, neither a coloration nor a brownish-black precipitate should develop within five minutes. (Indicating none present.)

Hydrochloric Acid (Chlorides). — 5 c.c. of glycerin diluted with 25 c.c. of water should exhibit at most a slight, opalescent turbidity on the addition of silver nitrate solution. (Indicating less than 0.0004 per cent Cl.)

Sulphuric and Oxalic Acids. — The solution of 5 c.c. of glycerin in 25 c.c. of water should not be affected by barium chloride solution or by calcium chloride solution. (Indicating less than 0.0024 per cent SO_3, and less than 0.0064 per cent $H_2C_2O_4$).

Heavy Metals. — On diluting 5 c.c. of glycerin with 25 c.c. of water and adding hydrogen sulphide water, no change should appear. (Indicating none present.)

Calcium. — 5 c.c. of glycerin dissolved in 25 c.c. of water should not be affected by the addition of ammonium oxalate solution. (Indicating less than 0.005 per cent Ca.)

Sugars. — 5 c.c. of glycerin mixed with 5 c.c. of water and 5 drops of diluted hydrochloric acid are warmed a few minutes on a boiling water-bath, then 3 drops of solution of copper sulphate and 1 c.c. of solution of sodium hydroxide are added. Upon further warming no pronounced separation of cuprous oxide should occur. (Indicating less than 0.04 per cent as saccharose.)

Readily Carbonizable Matter. — 5 c.c. of glycerin mixed with an equal volume of concentrated sulphuric acid, and allowed to stand one hour, should not become darker than yellow. (Indicating none present.)

Dextrose, Ammonium Compounds, and Organic Bodies Such as Occur in Unpurified Glycerin. — On heating on a water-bath 1 c.c. of glycerin with 1 c.c. of sodium hydroxide solution, no ammonia should be evolved (to be detected by means of moistened litmus paper) (indicating less than 0.003 per cent NH_3); neither should a color develop, nor an odor resembling that of glue. (Indicating no dextrose and organic bodies present.)

Fatty Acid Esters. — 50 c.c. of glycerin, 50 c.c. of water, and 10 c.c. of decinormal solution of potassium hydroxide are mixed and warmed 15 minutes on a boiling water-bath. After cooling, at least 4 c.c. of decinormal solution of hydrochloric acid should be required for neutralization, using phenolphthalein as indicator. (Indicating less than 0.115 per cent as ethyl butyrate.)

II

GLYCERIN, Sp.Gr. 1.23

A clear, colorless, almost odorless liquid of neutral reaction. Soluble in all proportions in water and in alcohol. Insoluble in ether and in chloroform. Its specific gravity is 1.225–1.235.

TESTS OF PURITY

The tests of purity are to be made in the manner described under Glycerin, Sp.Gr. 1.250.

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GUAIACIN ACCORDING TO SCHMITT *

Guaiacin is a brownish, amorphous powder, obtained by a special method from guaiac wood. It is soluble in alcohol, is colored blue by oxidizers, is far more sensitive than guaiac resin, and serves as an excellent reagent for oxidases. Guaiacin is used in the form of a 5 per cent alcoholic solution, which keeps well in completely filled, dark-colored, well-stoppered bottles.

HEMATEIN

$C_{16}H_{12}O_6$. Mol. Wt. 300.09

Hematein occurs in reddish-brown plates, presenting a yellowish-green metallic luster. 100 parts of water at 20° C. dissolve 0.06 part of hematein. It is difficultly soluble in ether and in alcohol, and is insoluble in chloroform and benzene; but it is soluble in ammonia water with brownish-violet color, and in dilute sodium hydroxide solution with a bright-red color.

HEMATOXYLIN

$C_{16}H_{14}O_6+3H_2O$. Mol. Wt. 356.16

Colorless, or pale yellow, tetragonal prisms, melting at 100° to 120° C. with loss of water. On slowly cooling a solution saturated at boiling heat, hematoxylin crystallizes at times in the form of rhombic crystals, containing one molecule of water of crystallization. Hematoxylin is but slightly soluble in cold water and in ether, but is more soluble in borax solution and in hot water, and is easily soluble in alcohol. On exposure to light it gradually acquires a red color, and then dissolves to form pale yellow solutions. In ammonia water hematoxylin dissolves and yields a purple solution. Hematoxylin is used as an indicator in 0.5 per cent alcoholic solution.

NOTE. — Regarding the use of hematoxylin as an indicator in the titration of akaloids, see C. Kippenberger, Z. anal. Chem., **39**, 201 (1900); *abst.*, J. Chem. Soc., **78**, II, 637 (1900). J. Messner, Z. angew. Chem., **16**, 444 (1903); 4 *abst.*, J. Chem. Soc., **84**, II, 519 (1903).

* Le Bois de Gajac, Thèse de Nancy, 1875. Report on the Advancements of Pharmaceutical Chemistry and Therapeutics, E. Merck's Annual Report on the year 1902, p. 75.

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HIDE POWDER

White or yellowish-white, woolly powder, prepared from the best quality of hide from which the hair has been removed with lime and which has been thoroughly washed. Hide powder should have but a slight odor and should be specially free from odors of decomposition products. It is used for tannin determination.

TEST OF PURITY

Determination of the Water-Soluble Constituents.* — Introduce 7 gm. of the hide powder into a bell-filter, as described by Proctor (see Lunge, Chem.-techn. Untersuch.-Meth., 5 ed., **3**, 715 (1905); Allen, Com. Org. Anal., 3 ed., Vol. III, Pt. I, p. 83 (1900)). Then suspend the filter in a beaker of about 150 to 200 c.c. capacity, fix the siphon, by means of a clamp, so that the bell-filter almost reaches the bottom of the beaker, and then pour into the beaker a small quantity of water in order to moisten the hide powder by capillarity. After the powder has become moist, which requires about an hour, the beaker is filled, and suction carefully applied to the siphon until the filtrate begins to drop slowly. The filtration of 90 to 100 c.c. requires about one and a half to two hours. The 30 c.c. first siphoned off are rejected; the next 50 c.c. are evaporated to dryness on the water-bath. The residue from the evaporation is then dried to constant weight in an air-bath at 100° to 105° C. The weight of this residue, conforming to the requirements laid down by the International Conference of Chemists for Leather Industries, should not exceed 0.005 gm.

HYDRAZINE SULPHATE

$(NH_2)_2H_2SO_4$. Mol. Wt. 130.14

Colorless crystals, soluble in 40 parts of cold, and in 8 parts of boiling water; insoluble in alcohol. The aqueous solution (1 : 50) is acid to litmus paper.

TESTS OF PURITY

Chlorides. — The solution of 1 gm. of hydrazine sulphate in 50 c.c. of water should not be more than slightly opalescent upon the addition of nitric acid and silver nitrate. (Indicating less than 0.002 per cent Cl.)

Heavy Metals. — The solution of 1 gm. of hydrazine sulphate in 50 c.c. of water, should remain unchanged upon addition of hydrogen sulphide water. The further addition of ammonia water and ammonium sulphide solution should produce neither a green or brown color nor a precipitate. (Indicating none present.)

Non-volatile Matter. — Upon volatilizing 1 gm. of hydrazine sulphate by heating, no weighable residue should remain. (Indicating less than 0.05 per cent.)

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* As the quantity of water-soluble constituents in the various hide powders varies greatly, it is always necessary, before using a hide powder for tannin determinations, to determine the quantity of soluble constituents by a blank test according to the method described here.

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Quantitative Determination. — Titrate 1 gm. of hydrazine sulphate, dissolved in 100 c.c. of water, with normal solution of potassium hydroxide, using methyl orange as indicator.

1 c.c. of normal KOH = 0.130138 gm. of $(NH_2)_2H_2SO_4$, log. 11440.

Dissolve 1 gm. of hydrazine sulphate in 100 c.c. of water. To 10 c.c. of this solution add 1 gm. sodium bicarbonate and titrate with decinormal solution of iodine, using starch solution as indicator.

1 c.c. of decinormal I = 0.00325345 gm. $(NH_2)_2H_2SO_4$, log. 51234.

HYDROGEN PEROXIDE, 30 PER CENT

(Perhydrol)

H_2O_2. Mol. Wt. 34.02

A colorless liquid of specific gravity 1.115 to 1.119, containing about 30 per cent by weight of hydrogen peroxide.* The solution is acid to litmus paper.†

TESTS OF PURITY

Free Acids. — Dilute 10 c.c. of hydrogen peroxide with 100 c.c. of water and then decompose it with small pieces of platinum black or manganese dioxide. Allow the liquid to stand with repeated shaking until the oxygen has escaped, filter, and to the filtrate add solution of phenolphthalein and decinormal solution potassium hydroxide until a pink color remains. Not more than one drop of the solution of potassium hydroxide should be required. (Indicating none present.)

Sulphuric Acid. — Dilute 1 c.c. of the hydrogen peroxide with 20 c.c. of water, add 1 c.c. of hydrochloric acid, heat to boiling, and add barium chloride solution. No precipitate of barium sulphate should form on standing fifteen hours. (Indicating less than 0.0075 per cent SO_3.)

Residue on Evaporation (Sulphuric and Phosphoric Acids, etc.). — 10 c.c. of the hydrogen peroxide, when heated on the water-bath, should completely volatilize and leave no weighable residue. (Indicating less than 0.0045 per cent.)

Oxalic Acid. — The solution of 2 c.c. of hydrogen peroxide in 10 c.c. of water should not become turbid on the addition of calcium chloride solution. Indicating less than 0.035 per cent $H_2C_2O_4$.)

Hydrochloric Acid. — The solution of 1 c.c. of hydrogen peroxide in 20 c.c. of water with 1 c c. of nitric acid should not be changed on the addition of a few drops of silver nitrate solution. (Indicating less than 0.0005 per cent Cl.)

Hydrofluoric Acid. — Evaporate to a small volume 10 c.c. of hydrogen peroxide mixed with 0.1 gm. of magnesium oxide, transfer the concentrated liquid to a watch glass, evaporate to dryness on the latter, pour concentrated sulphuric acid over the residue, and allow the whole to stand two to three hours in a warm place. After washing off the glass, no etching should be visible. (Indicating less than 0.005 per cent HF:)

Phosphoric Acid. — Concentrate 5 c.c. of hydrogen peroxide on the water-bath, take up any residue with 3 c.c. of water, and add 1 c.c. of magnesia mixture

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* The solution, containing 30 per cent, by weight, of hydrogen peroxide, is also designated as "100-volumes hydrogen peroxide," because this solution is capable of yielding 100 times its volume of free oxygen.

† The acid reaction is entirely due to the high content of hydrogen peroxide.

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and 3 c.c. of ammonia water. No precipitation should take place on standing fifteen hours. (Indicating less than 0.0006 per cent P_2O_5.)

Quantitative Determination. — Weigh off 1 gm. of hydrogen peroxide in a graduated flask of 100 c.c. capacity, and fill with water to the mark. Dilute 20 c.c. of this liquid with 50 c.c. of water, add 20 c.c. of 16 per cent sulphuric acid, and titrate with decinormal potassium permanganate. At least 35 c.c. should be required to produce the end-point.

1 c.c. of decinormal $KMnO_4 = 0.0017008$ gm. of H_2O_2, log. 23065.

HYDROGEN SULPHIDE WATER

A clear, colorless liquid, having a strong odor of hydrogen sulphide gas, and affording a voluminous precipitate of sulphur on the addition of ferric chloride solution. Hydrogen sulphide water has an acid reaction to litmus paper.

TEST OF PURITY

Iron. — Hydrogen sulphide water should not be changed upon the addition of ammonia water. (Indicating none present.)

HYDROXYLAMINE HYDROCHLORIDE

NH_2OH HCl. Mol. Wt. 69.50

Colorless crystals, soluble in 1.5 parts of water and in 15 parts of alcohol, and also in glycerin. The aqueous solution is acid to litmus paper.

TESTS OF PURITY

Non-volatile Matter. — 1 gm. of hydroxylamine hydrochloride, heated on platinum foil, should volatilize and leave no weighable residue. (Indicating less than 0.05 per cent.)

Ammonium Chloride. — The 1 : 20 alcoholic solution should afford no precipitate on adding platinic chloride solution. (Indicating less than 0.3 per cent.)

Sulphuric Acid. — On adding barium chloride solution to 20 c.c. of a 1 : 10 aqueous solution of hydroxylamine hydrochloride, no precipitate of barium sulphate should form within fifteen hours. (Indicating less than 0.0005 per cent SO_3.)

Heavy Metals. — The solution of 1 gm. of hydroxylamine hydrochloride in 20 c.c. of water should show scarcely any change upon the addition of hydrogen sulphide water. (Indicating at most a trace.)

Arsenic. — Upon thoroughly mixing 1 gm. of hydroxylamine hydrochloride and 3 c.c. of solution of tin chloride no dark color should appear within one hour. (Indicating less than 0.0015 per cent As.)

Quantitative Determination.— Dissolve 1 gm. of hydroxylamine hydrochloride in water and dilute to 100 c.c. After boiling 10 c.c. of this solution five minutes with 20 c.c. of a solution of ferric ammonium sulphate (saturated in the cold) and 10 c.c. of diluted sulphuric acid, dilute with 25 c.c. of boiled water and titrate with decinormal solution of potassium permanganate.

1 c.c. of decinormal $KMnO_4 = 0.003475$ gm. of NH_2OH HCl, log. 54095.

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INDIGO

I

SYNTHETIC INDIGO

A fine, dark-blue powder, containing at least 95 per cent of indigo blue.

TESTS OF PURITY

Ash. — 1 gm. of indigo upon ignition should leave a residue not exceeding 0.01 gm. in weight. (Indicating not more than 1 per cent.)

Moisture. — On drying 1 gm. of indigo at 100° C. to constant weight, the loss of weight should not exceed 0.01 gm. (Indicating not more than 1 per cent.)

Quantitative Determination of Indigo Blue (Indigotin). — Warm on a water-bath 0.5 gm. of finely powdered indigo in a beaker with 10 gm. concentrated sulphuric acid, frequently stirring until dissolved, and dilute to 1 liter. To 50 c.c. of the solution add 600 c.c. of water, and titrate with decinormal solution of potassium permanganate. The end-point is the change from green to light yellow, not the incipient red color.

1 c.c. of decinormal $KMnO_4$ = 0.007493 gm. of indigotin,* log. 87466.

II

VEGETABLE INDIGO

Dark-blue, light pieces, which, on being rubbed with a horn spoon, or other hard substance, exhibit a coppery luster. Vegetable indigo should contain at least 60 per cent of indigo blue.

TESTS OF PURITY

Ash. — 1 gm. of indigo should not leave more than 0.12 gm. of ash. (Indicating not more than 12 per cent.)

Moisture. — On drying 1 gm. of indigo to constant weight at 100° C., the loss in weight should not exceed 0.06 gm. (Indicating not more than 6 per cent.)

Quantitative Determination of Indigo Blue (Indigotin). — Warm on the water-bath one or two hours, 1 gm. of finely powdered indigo with 20 gm. of concentrated sulphuric acid, stirring thoroughly. Dilute to 1 liter, filter, and to 50 c.c. of the filtrate add 50 c.c. of water and 32 gm. of sodium chloride. After standing two hours, filter, wash the precipitate with saturated solution of sodium chloride, and, after dissolving the precipitate in hot water, add 500 c.c. of water containing 1 c.c. of sulphuric acid, and titrate with decinormal solution of potassium permanganate. The end-point is the change from green to light yellow, not the incipient red color.

1 c.c. of decinormal $KMnO_4$ = 0.007493 gm. indigotin,* log. 87466.

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* O. Miller & J. Smirnoff, Ber., **41**, 1366 (1908); *abst.*, J. Chem. Soc., **94, I,** 468 (1908).

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INDIGO SOLUTIONS

I

INDIGO SOLUTION, 1 : 40

4 gm. of synthetic indigo are heated upon the water-bath with 28 gm. of Acid Sulphuric, Fuming (8 to 10 per cent free SO_3), with frequent stirring until solution results. This solution after cooling is poured into 128 c.c. of water.

II

INDIGO SOLUTION, 1 : 1000

This solution is prepared by diluting 4 c.c. of Indigo Solution, 1 : 40, with 96 c.c. of water.

IODEOSIN

(Tetraiodofluorescein)

$C_{20}H_8I_4O_5$. Mol. Wt. 835.74

A scarlet-red powder, yielding with alcohol a deep-red, and with ether a yellowish-red, solution. Iodeosin is insoluble in water containing a trace of hydrochloric acid. The solution used as an indicator is prepared by dissolving 1 gm. of iodeosin in 100 c.c. of boiling alcohol (about 85 per cent by weight), diluting to 500 c.c. with alcohol, and filtering after standing twenty-four hours.

TEST OF SENSITIVENESS

Introduce 100 c.c. of water, containing 5 drops of the above iodeosin solution, into a colorless, glass-stoppered flask, and then overlay it with 30 c.c. of ether. Then run in by drops, from a burette, centinormal hydrochloric acid solution, vigorously shaking after the addition of each drop, until the aqueous layer just becomes colorless. Now add 5 drops more of the iodeosin solution. After shaking again, the aqueous layer must not acquire a pink color; or, should it do so, the color must disappear on the addition of 1 drop of centinormal hydrochloric acid solution.

NOTE. — Regarding the use of iodeosin for the estimation of alkaloids, see C. Kippenberger, Z. anal. Chem., **39**, 205 (1900); *abst.*, J. Chem. Soc., **78**, II, 637 (1900). Merck's Berichte, 9, 32 (1900); Merck's Annual Report, 13, 37 (for the year 1900).

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IODINE

(Iodine Resublimed)

I. Atomic Wt. 126.92

Blackish-gray, dry, rhombic plates or scales, with a metallic luster, easily soluble in alcohol, ether, and chloroform. Iodine dissolves in about 4500 parts of water, and is very freely soluble in an aqueous solution of potassium iodide. The preparation contains 100 per cent of I.

TESTS OF PURITY

Non-volatile Matter. — On cautiously heating 1 gm. of iodine in a porcelain dish, no weighable residue should remain. (Indicating less than 0.05 per cent.)

Cyanogen, Chlorine, and Bromine. — Shake 0.5 gm. of powdered iodine with 20 c.c. of water, and filter. To 10 c.c. of the filtrate, add, drop by drop, sulphurous acid until decolorized, then add a granule of ferrous sulphate, 1 drop of ferric chloride solution and 2 c.c. of sodium hydroxide solution. Warm to about 60° C., and add 10 c.c. of hydrochloric acid. The liquid should not acquire a blue color. (Indicating less than 0.05 per cent CN.) To the other 10 c.c. of the filtrate add 1 c.c. of ammonia water and 5 drops of silver nitrate solution, and again filter. On adding to the filtrate 2 c.c. of nitric acid no precipitate, and not more than an opalescent turbidity, should develop.* (Indicating less than 0.12 per cent Cl and Br as Cl.)

Quantitative Determination. — Dissolve 0.2 gm. of iodine, and 1 gm. of potassium iodide in 1 c.c. of water, add 50 c.c. of water, and titrate with decinormal solution of sodium thiosulphate.†

1 c.c. of decinormal $Na_2S_2O_3$ = 0.012692 gm. of I, log. 10352.

IODINE WATER

A saturated aqueous solution, containing about 0.2 gm. of iodine in 1000 c.c.

TEST OF STRENGTH

Titrate 100 c.c. of iodine water with decinormal sodium thiosulphate solution. 1 c.c. of decinormal $Na_2S_2O_3$ = 0.012692 gm. of I, log. 10352

IRON

Fe. Atomic Wt. 55.84

Metallic iron is used in the analytical laboratory in the form of thin, polished wire (piano wire), or gray powder obtained by reduc-

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* The filtrate contains traces of dissolved silver iodide; hence, on acidulating with nitric acid some opalescence always occurs.

† The titer of the decinormal sodium thiosulphate solution should be controlled by means of potassium biniodate.

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tion with hydrogen (iron by hydrogen), and also in the form of the ordinary gray, lustrous powdered iron.

I

IRON WIRE

(Piano Wire)

Iron wire serves chiefly for the standardization of potassium permanganate solutions. It was formerly believed, without further consideration, that the wire contained 99.6 to 99.8 per cent of pure, active iron. Treadwell, however, has shown that the various kinds of wire exhibit a decidedly variable value towards potassium permanganate solution, and that several exhibit a value corresponding to over 100 per cent of Fe, which statement is fully confirmed by Lunge. The explanation of this lies in the presence of carbon in the wire, principally as carbide of iron, which, upon dissolving in acid, becomes converted into hydrocarbons, exerting a marked reducing action on the permanganate.

Notwithstanding this, iron wire is a convenient, unalterable, and also perfectly reliable substance for standardizing permanganate solutions, if we have once ascertained its effective value compared with potassium permanganate solution the titer of which has been accurately determined by means of some other standard.

For a satisfactory means of fixing the titer of permanganate solution, sodium oxalate may be recommended (see Sodium Oxalate, according to Sörensen). Or* starting with sodium bicarbonate, by drying the latter at about 300° C., thus obtaining sodium carbonate, preparing a standard solution of this, from this preparing a solution of fifth-normal hydrochloric acid, from this a solution of fifth-normal potassium hydroxide, from this a solution of oxalic acid, and finally a solution of the permanganate.† Where a gas volumeter is at hand, the titer may also be ascertained with hydrogen peroxide,

* G. Lunge, Z. angew. Chem., **17**, 267 (1904); *abst.*, J. Chem. Soc., **86**, II, 289 (1904).

† For titrations in hydrochloric acid solutions, use a titer obtained in the same way. The oxalic acid titer is, according to Reinhardt, not suitable for the iron titration.

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according to the nitrometer method. (G. Lunge, Chem.-tech. Untersuch.-Meth., 5 ed., 1, 127, (1904). J. Chem. Soc., **58**, 1468 (1890).

II

IRON BY HYDROGEN

(Reduced Iron)

A gray, lusterless, fine powder, containing at least 90 per cent of Fe.

TESTS OF PURITY

Solubility in Sulphuric Acid. — 10 gm. of reduced iron are dissolved in a mixture of 20 c.c. of sulphuric acid (sp.gr. 1.84) and 200 c.c. of water. The insoluble residue should be filtered off, and when dried at 100° C., its weight should not exceed 0.05 gm. (Indicating not more than 0.5 per cent insoluble matter.)

Sulphides. — Pour a mixture of 10 c.c. of hydrochloric acid and 10 c.c. of water over 1 gm. of reduced iron in a test-tube. Filter paper moistened with solution of lead acetate should not be darkened in color on exposure for one minute to the liberated hydrogen. (Indicating less than 0.007 per cent S.)

Sodium Carbonate. — On shaking 5 gm. of reduced iron with 50 c.c. of water and filtering, the filtrate should not turn red litmus paper blue; and on evaporation it should leave not more than 0.003 gm. of residue. (Indicating not more than 0.06 per cent.)

Nitrogen. — Dissolve 20 gm. of reduced iron in a mixture of 20 c.c. of concentrated sulphuric acid (sp.gr. 1.84) and 200 c.c. of water, with the aid of heat. Allow to cool, and when cold add 100 c.c. of nitrogen-free sodium hydroxide solution I, and distil off about 75 c.c., collecting the distillate in a receiver containing about 10 c.c. of water and 2 to 3 c.c. of fifth-normal hydrochloric acid. Titrate the distillate with fifth-normal potassium hydroxide solution, using methyl orange as indicator. The ammonia from the iron should not neutralize more than 0.2 c.c. of the acid. (Indicating not more than 0.0028 per cent N.)

Arsenic. — Pour 10 c.c. of hydrochloric acid over a mixture of 1 gm. of reduced iron and 1 gm. of potassium chlorate, and when the reaction has subsided, heat until all free chlorine had been expelled, and filter. On now adding 15 c.c. of stannous chloride solution to 5 c.c. of the filtrate, a dark coloration should not develop within one hour. (Indicating less than 0.0015 per cent As.)

Quantitative Determination. — Introduce into a graduated flask of 100 c.c. capacity 1 gm. of reduced iron, 10 gm. of finely powdered mercuric chloride, and 50 c.c. of boiling water. Place the flask on wire gauze, heat over a small flame, boil about five minutes with frequent shaking, then immediately fill the flask to the mark with cold, boiled water. Cool the mixture to 15° C., add water to the mark, shake thoroughly, and, tightly stoppering the flask, set it aside to deposit. Now filter, and to 10 c.c. of the filtrate add immediately 10 c.c. of 16 per cent sulphuric acid, and titrate with decinormal potassium permanganate until a faint red color persists. At least 16.2 c.c. of the permanganate solution should be required.

After the pink color has been destroyed by the addition of solution of tartaric acid, dissolve in the liquid 2 gm. of potassium iodide, allow to stand two hours

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in the stoppered flask at room temperature, and titrate with decinormal sodium thiosulphate solution, using starch solution as the indicator.

1 c.c. of decinormal $KMnO_4$ or 1 c.c. of decinormal $Na_2S_2O_3 = 0.005584$ gm. of Fe, log. 74695.

III

IRON POWDER

A fine, heavy, gray powder with metallic luster, and containing at least 97.7 per cent of Fe.

TESTS OF PURITY

Solubility in Sulphuric Acid. — 10 gm. of powdered iron are dissolved in a mixture of 20 c.c. of concentrated sulphuric acid (sp.gr. 1.84) and 200 c.c. of water. The insoluble residue should be filtered off through a filter previously dried at 100° C. and weighed; the filter and residue are then washed, and dried at 100° C. and weighed. The weight of the residue should not exceed 0.01 gm. (Indicating not more than 0.1 per cent insoluble matter.)

Nitrogen and Arsenic. — These tests are carried out as detailed under Iron by Hydrogen.

Foreign Heavy Metals. — Dissolve 1 gm. of powdered iron in 10 c.c. of hydrochloric acid mixed with 5 c.c. of nitric acid, and heat the solution until it has a brown color. Dilute the solution with 35 c.c. of water, add 30 c.c. of ammonia water, and filter. The filtrate should not have a blue color; nor should it show any change on the addition of hydrogen sulphide water. (Indicating none present.)

Quantitative Determination. — Dissolve 1 gm. of powdered iron in about 50 c.c. of dilute sulphuric acid, and dilute the solution to 100 c.c. To 10 c.c. of this solution add decinormal potassium permanganate solution until the liquid has a slight red color, and, after destroying the red color by the addition of tartaric acid solution, add 2 gm. of potassium iodide. Allow the mixture to stand two hours in a closed flask at room temperature, and then titrate with decinormal solution of sodium thiosulphate with starch solution as indicator. At least 17.5 c.c. should be required to combine with the liberated iodine.

1 c.c. of decinormal $Na_2S_2O_3 = 0.005584$ gm. of Fe, log. 74695.

IRON CHLORIDE, FERRIC

$FeCl_3 + 6H_2O$. Mol. Wt. 270.31

Yellow, crystalline masses, very deliquescent in air. Ferric chloride is easily soluble in water, alcohol, and a mixture of alcohol and ether. The solutions are acid to litmus paper.

TESTS OF PURITY

Basic Salt and Other Matter Difficultly Soluble in Water. — 10 gm. of ferric chloride should completely dissolve in 10 c.c. of water, and yield a perfectly clear solution. (Indicating none present.)

Hydrochloric Acid and Chlorine. —On boiling 10 c.c. of a solution (1 : 1) of ferric chloride for 2 or 3 minutes in a test-tube capped with blue litmus paper, the paper should not be reddened. (Indicating less than 0.35 per cent HCl.) In

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a similar test employing zinc iodide-starch paper, the latter should not be turned blue. (Indicating less than 0.001 per cent Cl.)

Arsenic. — A mixture of 1 c.c. of ferric chloride solution (1 : 1) and 3 c.c. of stannous chloride solution should not acquire a darker color within one hour. (Indicating less than 0.0011 per cent As.)

Ferrous Salt. — Add 1 c.c. of hydrochloric acid and a few drops of potassium ferricyanide solution to the 1 : 20 aqueous solution of ferric chloride; no blue color should develop. (Indicating less than 0.0025 per cent Fe″.)

Zinc, Copper, Nitric Acid, etc. (Alkali Salts, Calcium). — Dilute 20 c.c. of ferric chloride solution (1 : 1) with 100 c.c. of water, add 30 c.c. of ammonia water, filter, and bring the filtrate to a volume of 75 c.c. by washing. On evaporating 50 c.c. of the filtrate, which must be colorless, and igniting the residue, the weight of the latter should not exceed 0.001 gm. (Indicating not more than 0.0117 per cent alkali salts, calcium, etc.) On mixing 2 c.c. of the filtrate with 2 c.c. of concentrated sulphuric acid, and overlaying this mixture with 1 c.c. of ferrous sulphate solution, no brown zone should form at the contact-surfaces of the two liquids. (Indicating less than 0.025 per cent N_2O_5.) The remainder of the filtrate acidulated with acetic acid and treated with potassium ferrocyanide solution should show neither a white nor a brownish-red precipitate upon standing two hours. (Indicating less than 0.0015 per cent Zn and less than 0.005 per cent Cu.)

Sulphates. — Dissolve 10 gm. of ferric chloride in 100 c.c. of water, add 25 c.c. of ammonia water, filter, acidulate the filtrate with acetic acid, and add barium chloride solution. No precipitate of barium sulphate should form on standing fifteen hours. (Indicating less than 0.0025 per cent SO_3.)

Quantitative Determination. — Dissolve 1 gm. of ferric chloride in 100 c.c. of water, add 2 gm. of potassium iodide and 5 c.c. of hydrochloric acid. After allowing the mixture to stand in a well-closed flask for about two hours, titrate the free iodine with decinormal solution of sodium thiosulphate.

1 c.c. of decinormal $Na_2S_2O_3 = 0.016222$ gm. $FeCl_3$, log. 21010, or 0.0270315 gm. $FeCl_3 + 6H_2O$, log. 43187.

IRON CHLORIDE, FERRIC, SOLUTION

A clear, deep yellowish-brown liquid, of specific gravity 1.280 to 1.282. 100 parts contain 10 parts of Fe, = 29 parts of $FeCl_3$.

TESTS OF PURITY

The tests to be made are those given under Iron Chloride, Ferric. But, for each gram of the crystallized ferric chloride, 2 gm. of the solution of ferric chloride are to be taken.

IRON CHLORIDE, FERROUS

$FeCl_2 + 4H_2O$. Mol. Wt. 198.82

A pale-green hygroscopic powder, soluble in an equal weight of water acidulated with a few drops of hydrochloric acid. Ferrous chloride is also soluble in alcohol.

TESTS OF PURITY

Oxychloride. — The solution of 1 gm. of ferrous chloride in 1 c.c. of water and 2 to 3 drops of hydrochloric acid should be pale green or green in color, and

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should not have any yellowish-green tint. On adding hydrogen sulphide water, the solution should show only a very slight turbidity, due to the separation of sulphur. (Indicating at most a small amount.*)

Sulphates, Copper, Zinc, and Salts of the Alkalies. — Heat a solution of 5 gm. of ferrous chloride in 10 c.c. of water and 5 c.c. of nitric acid (sp.gr. 1.3) until brown in color, dilute to 120 c.c., add 30 c.c. of ammonia water, filter, and bring the filtrate to a volume of 75 c.c. by washing. The filtrate should be colorless. Evaporate 50 c.c. of the filtrate and ignite the residue. The weight of the latter should not exceed 0.001 gm. (Indicating not more than 0.03 per cent alkali salts.) Acidulate 20 c.c. of the filtrate with acetic acid and add barium chloride solution. No cloudiness should appear within five minutes. (Indicating less than 0.0075 per cent SO_3.) The remainder of the filtrate acidified with acetic acid should show neither a white nor a brownish-red precipitate upon addition of potassium ferrocyanide solution, followed by two hours' standing. (Indicating less than 0.0075 per cent Zn and less than 0.03 per cent Cu.)

Arsenic. — The solution of 1 gm. of ferrous chloride in 1 c.c. of water acidulated with a few drops of hydrochloric acid, should not acquire a darker color on adding 3 c.c. of stannous chloride solution and standing one hour. (Indicating less than 0.0015 per cent As.)

IRON OXIDE

Brandt's Ferric Oxide †

Fe_2O_3. Mol. Wt. 159.68

A reddish-brown powder soluble in concentrated hydrochloric acid. The preparation is used as a starting material for making standardized solutions of potassium permanganate, especially for the determination of iron in hydrochloric acid solution. It should be dried at 120° C. immediately before using.

TESTS OF PURITY

Water and Volatile Substances. — The iron oxide is dried to constant weight at 120° C. 3 gm. of this dry material should not lose more than 0.003 gm. in weight upon ignition. (Indicating not more than 0.1 per cent.) Avoid the contact of the reducing flame with the platinum crucible.

Substances Soluble in Water. — Heat to boiling 100 c.c. of water with 5 gm. of the iron oxide and then filter. 50 c.c. of the filtrate, which must be perfectly clear, should leave no weighable residue upon evaporation followed by ignition. (Indicating less than 0.01 per cent.)

Chlorides. — Fuse in a platinum crucible a mixture of 1 gm. of the iron oxide and 3 gm. of anhydrous sodium carbonate. After cooling, leach the melt with 50 c.c. of hot water, filter the solution, acidulate the filtrate with 20 c.c. of nitric acid, and add silver nitrate solution. At most only an opalescence may appear. (Indicating less than 0.005 per cent Cl.)

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* Small amounts of iron oxychloride are always present in ferrous chloride.

† L. Brandt, Chem., Ztg., **32**, 812, 830, 840, 851 (1908); *abst.*, J. Chem. Soc., **94**, II, 899 (1908).

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Nitrates. — Heat to boiling a mixture of 5 c.c. of 30 per cent acetic acid and 10 c.c. of water with 1 gm. of the iron oxide, filter, and to the filtrate add 1 drop of 1 : 1000 indigo solution and 10 c.c. of concentrated sulphuric acid. The blue color of the fluid should not disappear on shaking. (Indicating less than 0.0032 per cent N_2O_5.)

Sulphates. — A mixture of 1 gm. of the iron oxide with a solution of 4 gm. crystallized sodium carbonate in 20 c.c. of water is heated one hour on a boiling water-bath with repeated shaking, and then filtered. The filtrate, after the addition of 10 c.c. of hydrochloric acid, is heated to boiling and mixed with barium chloride solution. After standing two hours there should be no separation of barium sulphate. (Indicating less than 0.035 per cent SO_3.)

Silicates. — Fuse in a platinum crucible a mixture of 3 gm. of the iron oxide and 15 gm. of anhydrous sodium carbonate, leach the cooled melt with hot water, and filter the solution. Acidulate the filtrate with hydrochloric acid, evaporate it in a platinum dish, dry the residue one-half hour at about 120° C., and then dissolve it in 10 c.c. of hydrochloric acid, and 50 c.c. of water. Filter the solution, wash with water, and incinerate the filter. The weight of the ash should be not more than 0.001 gm. (Indicating not more than 0.033 per cent SiO_2.)

Ferrous Oxide. — Dissolve 1 gm. of the iron oxide by warming on the water-bath with 10 c.c. of fuming hydrochloric acid, and dilute the solution with 40 c.c. of water. On mixing this solution with potassium ferricyanide solution, neither a green nor a blue color should appear. (Indicating less than 0.003 per cent.)

Substances Insoluble in Hydrochloric Acid, Foreign Heavy Metals, Lime and Magnesia.—Dissolve 10 gm. of the iron oxide in 70 c.c. of fuming hydrochloric acid by warming on the water-bath. The solution, diluted with 200 c.c. of water, should be perfectly clear and should contain no, or only an exceedingly slight, insoluble residue. In the latter case filter the solution, wash the residue, first with diluted hydrochloric acid and then with water, ignite and weigh. The weight of the residue, insoluble in hydrochloric acid, should not exceed 0.001 gm. (Indicating not over 0.01 per cent insoluble substances.)

Pass hydrogen sulphide gas into the filtrate until the ferric chloride is fully reduced; the separated sulphur should not be dark-colored. Filter the fluid. Warm the sulphur in a porcelain dish on the water-bath with fuming hydrochloric acid and filter the solution after diluting it with water. Ignite the residue together with the filter in the porcelain dish, dissolve the ash in hydrochloric acid, and combine this solution with the hydrochloric acid extract. On passing into this solution hydrogen sulphide gas, neither a coloration nor the separation of a precipitate should occur. (Indicating no foreign heavy metals present.)

Boil the filtrate obtained after precipitation with hydrogen sulphide until the hydrogen sulphide is expelled, then oxidize with nitric acid and add an excess of ammonia water. Warm the solution until the odor of ammonia has almost disappeared, filter, and wash the precipitate with hot water. Dissolve a part of the precipitate of ferric hydroxide, about half, in hydrochloric acid, and after diluting with water, precipitate hot with sodium hydroxide solution (30 gm. of sodium hydroxide, from sodium, in 70 c.c. of water) in a platinum dish and filter. Acidulate the filtrate with hydrochloric acid, add ammonia water to alkaline reaction, and warm until the odor of ammonia has disappeared. No, or only an exceedingly slight, flocculent precipitate should be perceptible. In the latter case, filter, wash, and ignite the precipitate. The weight of the residue should not exceed 0.001 gm. (Indicating not more than about 0.02 per cent Al_2O_3.)

Fuse a small portion of the precipitate of ferric hydroxide with anhydrous sodium carbonate in the air (upon the cover of a platinum crucible); the melt should not have a green color. (Indicating no Cr present.)

Mix the filtrate obtained after precipitation of the iron, with a little ammonium sulphide solution; neither a dark color nor a precipitate should appear. (Indi-

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cating no Co, Ni, etc. present.) After acidulating the fluid with hydrochloric acid, concentrate, filter, evaporate to dryness, and gently ignite the residue. After complete volatilization of the ammonium salts, dissolve the residue in hydrochloric acid, filter the solution, make alkaline with ammonia water, and add ammonium oxalate solution. Upon standing fifteen hours no, or only an exceedingly slight, separation of calcium oxalate should occur. In the latter case filter, wash, and ignite the precipitate. The weight of the calcium oxide should be not more than 0.001 gm. (Indicating not more than 0.007 per cent Ca.)

The fluid tested with ammonium oxalate solution should exhibit no precipitate within fifteen hours after the addition of ammonium phosphate solution. (Indicating less than 0.0002 per cent Mg.)

Quantitative Determination. — Dissolve 0.5 gm. of the iron oxide, which has been dried at 120° C., by warming on the water-bath with 10 c.c. of fuming hydrochloric acid and a few drops of nitric acid. Dilute the solution with 200 c.c. of water, heat to boiling, and add 20 c.c. of ammonia water. Filter off the precipitate, wash free from chlorides with hot water, dry, ignite, and weigh.

IRON SULPHATE, FERROUS

$FeSO_4+7H_2O$. Mol. Wt. 278.02

Pale, greenish-blue, monoclinic crystals, soluble in 1.8 parts of cold water and in 0.5 part of boiling water; insoluble in alcohol and in ether.

TESTS OF PURITY

Substances Insoluble in Water (Basic Iron Sulphate). — The 1 : 20 solution, freshly prepared with water which has previously been thoroughly boiled and then cooled, should be clear and have a greenish-blue color. (Indicating none present.)

Salts of the Alkalies, Copper, Zinc. — Add 5 c.c. of nitric acid (sp.gr. 1.3) to a solution of 5 gm. of ferrous sulphate in 100 c.c. of water, and boil for several minutes; then add 25 c.c. of ammonia water, filter, and bring the filtrate to a volume of 75 c.c. by washing. The filtrate should be colorless. The ignited residue, from evaporation of 50 c.c. of the filtrate, should not exceed 0.001 gm. in weight. (Indicating not more than 0.03 per cent alkali salts.) The remainder of the filtrate acidulated with acetic acid, treated with solution of potassium ferrocyanide, and allowed to stand two hours, should show neither a white nor a brownish-red precipitate. (Indicating less than 0.0036 per cent Zn and less than 0.012 per cent Cu.)

Quantitative Determination. — Dissolve 1 gm. of ferrous sulphate in 50 c.c. of well-boiled water, add 10 c.c. of concentrated sulphuric acid, and titrate with decinormal solution of potassium permanganate.

1 c.c. of decinormal $KMnO_4$ = 0.027802 gm. of $FeSO_4+7H_2O$, log. 44407.

IRON SULPHIDE

(Ferrous Sulphide)

FeS. Mol. Wt. 87.91

Dark gray or grayish-black, heavy, hard lumps or sticks, soluble in diluted hydrochloric or sulphuric acid with the copious evolution of hydrogen sulphide.

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TEST OF PURITY

Quantitative Determination. — Place 0.5 gm. of the finely powdered iron sulphide in a retort in the tubule of which is fixed a funnel-tube provided with a glass cock. After connecting the retort with a receiver containing 50 to 100 c.c. of decinormal iodine solution, allow to flow into the retort, through the funnel-tube, a mixture of 20 c.c. of water and 20 c.c. of dilute sulphuric acid; close the stop-cock, and heat to boiling. After the decomposition of the iron sulphide is complete, and the hydrogen sulphide has been entirely driven off (the iodine solution must not be fully decolorized), determine the excess of iodine by means of decinormal solution of sodium thiosulphate.

1 c.c. of decinormal I = 0.0043955 gm. of FeS, log. 64301.

IRON AND AMMONIUM SULPHATE, FERRIC

(Ferric Ammonium Sulphate; Ammonio-Ferric Alum)

$FeNH_4(SO_4)_2+12H_2O$. Mol. Wt. 482.22

Pale violet (amethyst colored), transparent, octahedral crystals, soluble in 2 parts of water and insoluble in alcohol. The aqueous solution has an acid reaction.

TESTS OF PURITY

Ferrous Salt. — Dissolve 1 gm. of ferric ammonium sulphate in 20 c.c. of water, and add 1 c.c. of hydrochloric acid and 1 drop of a freshly-prepared solution of potassium ferricyanide. Neither a green nor a blue color should develop. (Indicating less than 0.0025 per cent Fe''.)

Chlorides. — 30 c.c. of the 1 : 20 aqueous solution should undergo no change on the addition of 3 c.c. of nitric acid and silver nitrate solution. (Indicating less than 0.0003 per cent Cl.)

Zinc and Copper. — To a solution of 2 gm. of ferric ammonium sulphate in 50 c.c. of water add 10 c.c. of ammonia water and filter. The filtrate should be colorless, and upon acidulating with acetic acid, adding solution of potassium ferrocyanide, and allowing to stand two hours, neither a white nor a brownish-red precipitate should form. (Indicating less than 0.003 per cent Zn and less than 0.01 per cent Cu.)

Salts of the Alkalies. — To a solution of 5 gm. of ferric ammonium sulphate in 100 c.c. of water add 15 c.c. of ammonia water, filter, and evaporate the filtrate. The residue ignited should not exceed 0.002 gm. in weight. (Indicating not more than 0.04 per cent.)

Quantitative Determination. — Dissolve 1 gm. of ferric ammonium sulphate in 100 c.c. of water, add 2 gm. of potassium iodide and 5 c.c. of hydrochloric acid, allow to stand two hours in a tightly closed flask and then titrate the free iodine with decinormal solution of sodium thiosulphate.

1 c.c. of decinormal $Na_2S_2O_3 = 0.0482225$ gm. $FeNH_4(SO_4)_2+12H_2O$, log.68325.

IRON AND AMMONIUM SULPHATE, FERROUS

(Ferrous Ammonium Sulphate; Mohr's Salt)

$FeSO_4(NH_4)_2SO_4+6H_2O$. Mol. Wt. 392.16

Pale, bluish-green crystals, or light, bluish-green crystalline powder, soluble in 6 parts of water. Ferrous ammonium sulphate

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contains exactly one-seventh of its weight of Fe in the ferrous condition.

TESTS OF PURITY

Ferric Salts. — The solution of 1 gm. of powdered ferrous ammonium sulphate in 20 c.c. of boiled water with 1 c.c. of hydrochloric acid, should not afford an immediate red color on adding a few drops of potassium sulphocyanate solution. (Indicating less than 0.0008 per cent Fe'''.)

Copper, Zinc, and Alkali Salts. — Dissolve 5 gm. of ferrous ammonium sulphate in 50 c.c. of water, add 5 c.c. of nitric acid (sp.gr. 1.3), and boil the solution for a few minutes; then add to the liquid 15 c.c. of ammonia water, filter, and bring the filtrate to a volume of 50 c.c. by washing. The filtrate must be colorless.

Evaporate 30 c.c. of the filtrate on the water-bath to dryness and ignite. No weighable residue should remain. (Indicating less than 0.0167 per cent alkali salts.)

The remainder of the filtrate is acidulated with acetic acid and then solution of potassium ferrocyanide is added. Neither a white nor brownish-red precipitate should appear on standing for two hours. (Indicating less than 0.003 per cent Zn and less than 0.01 per cent Cu.)

Quantitative Determination. — Dissolve 1 gm. of ferrous ammonium sulphate in 50 c.c. of well-boiled and cooled water, add 10 c.c. of diluted sulphuric acid, and titrate with decinormal potassium permanganate solution.

1 c.c. of decinormal $KMnO_4$=0.039216 gm. of $FeSO_4(NH_4)_2SO_4+6H_2O$, log. 59347.

LACMOID

(Resorcinol Blue)

Lustrous, dark-violet scales soluble in alcohol, acetone, and ether, and but slightly soluble in water. The solution to be used as an indicator is prepared by dissolving 0.5 gm. of lacmoid in 100 c.c. of about 85 per cent (by weight) alcohol, and diluting the solution with 100 c.c. of water.

TEST OF SENSITIVENESS

Add 0.2 c.c. of the above lacmoid solution to 100 c.c. of distilled water freed from carbon dioxide by prolonged boiling in a platinum dish; the blue color of the water must change to red on the addition of 0.05 c.c. of decinormal solution of hydrochloric acid, and the red color should again become blue on the further addition of 0.05 c.c. of decinormal solution of potassium hydroxide.

NOTE. — Regarding the use of lacmoid for the titration of alkaloids see C. Kippenberger, Z. anal. Chem., **39**, 214 (1900); *abst.*, J. Chem. Soc., **78**, II, 637 (1900); J. Messner, Z. angew. Chem., **16**, 444, (1903); *abst.*, J. Chem. Soc., **84**, II, 519 (1903).

LEAD ACETATE

$Pb(C_2H_3O_2)_2+3H_2O$. Mol. Wt. 379.20

Colorless, translucent, gradually efflorescing crystals soluble in

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2.3 parts of water. On dissolving in alcohol decomposition takes place.

TESTS OF PURITY

Earths and Alkalies. — Dissolve 10 gm. of lead acetate in about 200 c.c. of water, and precipitate the lead from the solution with hydrogen sulphide gas; filter, evaporate the filtrate, and ignite the residue. The weight of the latter should then not exceed 0.002 gm. (Indicating not more than 0.02 per cent.)

Copper, Iron and Aluminum. — Dissolve 2 gm. of lead acetate in 10 c.c. of water, mix the solution with 3 c.c. of concentrated sulphuric acid, and filter. On adding an excess of ammonia water to the filtrate neither a precipitate nor a blue color should appear. (Indicating less than 0.001 per cent Cu, less than 0.002 per cent Fe, and less than 0.1 per cent Al.)

Lead Carbonate and Substances Insoluble in Water. — The solution of 5 gm. of lead acetate in 50 c.c. of freshly boiled water should be clear, or only very slightly opalescent. (Indicating at most a trace.)

Chlorides. — The 1 : 30 aqueous solution acidified with nitric acid should show no change with silver nitrate solution. (Indicating less than 0.0005 per cent Cl.)

Nitrates. — To the solution of 1 gm. of lead acetate in 10 c.c. of water are added 1 drop of a 1 : 1000 indigo solution and 10 c.c. of concentrated sulphuric acid; the blue color of the fluid should not disappear on agitation. (Indicating less than 0.0032 per cent N_2O_5.)

LEAD CHROMATE

$PbCrO_4$. Mol. Wt. 323.1

A yellowish-brown powder, or brown lumps, insoluble in water, and ammonia water; but almost completely soluble in solutions of the fixed alkali hydroxides, and, with decomposition, almost wholly soluble also in concentrated nitric acid.

TESTS OF PURITY

Substances Soluble in Water. — Shake 5 gm. of lead chromate in fine powder with 50 c.c. of warm water (at about 50° C.) for five minutes, then filter, evaporate the filtrate, and ignite the residue. The weight of the latter should not exceed 0.001 gm. (Indicating not more than 0.02 per cent.)

Organic Substances. — Lead chromate should evolve no carbon dioxide on ignition. (Indicating none present.)

NOTE.—Lead chromate which is to be used in the elementary analysis of organic compounds should first be gently ignited in a current of oxygen, as according to C. H. L. Ritthausen, J. prakt. Chem., N. F., **25**, 141 (1882); *abst.*, J. Chem. Soc., **42**, 898 (1882), when ignited in air, it retains carbon, which can only be burned off by ignition in a current of oxygen.

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LEAD OXIDE, BROWN

(Lead Super-, Di-, or Peroxide)

PbO_2. Mol. Wt. 239.1

I

LEAD OXIDE, BROWN, FREE FROM MANGANESE

A dark-brown, amorphous powder, insoluble in water. The preparation contains at least 95 per cent of PbO_2.

TESTS OF PURITY

Chlorides. — Boil 5 gm. of lead peroxide with 60 c.c. of water and 5 c.c. of nitric acid, filter, and to 30 c.c. of the filtrate add silver nitrate solution. At most a slight opalescent turbidity should appear. (Indicating less than 0.001 per cent Cl.)

Sulphates. — Digest 5 gm. of lead peroxide with 30 c.c. of a cold saturated aqueous solution of sodium bicarbonate for three or four hours, shaking frequently. Then filter, acidulate the filtrate with hydrochloric acid, boil the solution for ten minutes, and add 2 c.c. of barium chloride solution. No precipitate of barium sulphate should form on standing fifteen hours. (Indicating less than 0.0015 per cent SO_3.)

Substances Soluble in Water (Lead Nitrate, etc.). — Boil 2 gm. of lead peroxide with 60 c.c. of water, and filter; 40 c.c. of the filtrate should not leave a weighable residue upon evaporation and ignition. (Indicating less than 0.0375 per cent.)

Calcium and Alkalies. — Dissolve 2 gm. of lead peroxide in 25 c.c. of hydrochloric acid and 200 c.c. of water, boil ten minutes to expel the chlorine, and supersaturate with hydrogen sulphide gas. Filter off the precipitate, evaporate the filtrate, ignite and weigh the residue. The weight should not exceed 0.01 gm. (Indicating not more than 0.5 per cent.)

Manganese. — Heat 5 gm. of lead peroxide with 10 c.c. of concentrated sulphuric acid until completely decomposed. When cold, mix the mass with 20 c.c. of water and add 0.5 gm. of lead peroxide. On now warming again, the liquid should not acquire a red color. (Indicating less 0.0002 per cent Mn.)

Quantitative Determination. — Gently heat 0.5 gm. of lead peroxide in a porcelain dish with 50 c.c. of decinormal solution of oxalic acid and 25 c.c. of nitric acid until complete decomposition has taken place. Now dilute with 200 c.c. of water, heat the solution to the boiling-point, and allow decinormal potassium permanganate solution to run into it until the red color imparted by 1 drop of the permanganate solution no longer completely disappears within half a minute.

1 c.c. of decinormal $H_2C_2O_4 = 0.011955$ gm. of PbO_2, log. 07755.

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II

LEAD OXIDE, BROWN, FOR DENNSTEDT'S ULTIMATE ANALYSIS

A dark-brown, amorphous powder, insoluble in water, used for ultimate organic analyses according to Dennstedt.*

TESTS OF PURITY

Chlorides, Calcium, and Alkalies. — The tests are to be carried out as detailed under Lead Oxide, Brown, Free from Manganese.

Sulphates. — Digest 25 gm. of lead peroxide with 50 c.c. of 20 per cent aqueous solution of sodium carbonate for one hour on the water-bath, shaking frequently. Then filter, wash the residue with 50 c.c. of hot water, acidulate the filtrate with hydrochloric acid, boil the solution for ten minutes, and add 2 c.c. of barium chloride solution. No precipitate of barium sulphate should form on standing fifteen hours. (Indicating less than 0.0008 per cent SO_3.)

Nitrates. — Boil 1 gm. of lead peroxide with a mixture of 5 c.c. of diluted acetic acid and 10 c.c. of water, filter, and color the filtrate blue by adding 1 drop of a 1 : 1000 indigo solution. The blue color should not disappear on the addition of 10 c.c. of concentrated sulphuric acid and subsequent agitation. (Indicating less than 0.0032 per cent N_2O_5.)

Carbonates. — On pouring nitric acid over 5 gm. of lead peroxide, no evolution of gas should be seen, even when observed through a magnifying glass. (Indicating none present.)

Quantitative Determination. — This is to be carried out as detailed under Lead Oxide, Brown, Free from Manganese.

III

LEAD OXIDE, BROWN, GRANULATED

Lead peroxide intended for use in elementary analysis, prepared according to the directions of Prof. F. Pregl. †

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* Literature: M. Dennstedt, Z. anal. Chem., **41**, 525 (1902); *abst.*, J. Chem. Soc., **84**, II, 103 (1903); also: Anleitung zur vereinfachten Elementaranalyse, von Prof. Dr. M. Dennstedt, 1903. Published by O. Meissner, Hamburg. M. Dennstedt, Chem. Ztg., **28**, 35 (1904); *abst.*, J. Chem. Soc., **88**, II, 202 (1905). H. Hermann, Z. anal. Chem., **44**, 686 (1905); *abst.*, J. Chem. Soc., **88**, II, 767 (1905). M. Dennstedt, Z. anal. Chem., **45**, 26 (1906); *abst.*, J. Chem. Soc., **90**, II, 398 (1906). H. Hermann, Z. anal. Chem., **45**, 236 (1906); *abst.*, J. Chem. Soc., **90**, II, 200 (1906). M. Dennstedt, Chem. Ztg., **33**, 769 (1909); *abst.*, J. Chem. Soc., **96**, II, 759 (1909). E. Müller and B. Diethelm, Z. angew. Chem., **33**, 2118 (1910); *abst.*, J. Chem. Soc., **98**, II, 1110 (1910). M. Dennstedt, Ber., **41**, 600 (1908); *abst.*, J. Chem. Soc., **94**, II, 321 (1908). M. Dennstedt and F. Hassler, Chem. Ztg., **33**, 133 (1909); *abst.*, J. Chem. Soc., **96**, II, 270 (1909). H. Weil, Ber., **43**, 149 (1910); *abst.*, J. Chem. Soc., **98**, II, 242 (1910). M. Dennstedt and F. Hassler, Ber., **43**, 1197 (1910); *abst.*, J. Chem. Soc., **98**, II, 547 (1910).

† F. Pregl, Ber., **38**, 1434 (1905); *abst.*, J. Chem. Soc., **88**, 420 (1905).

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LEAD OXIDE, YELLOW

(Litharge)

PbO. Mol. Wt. 223.10

A yellow or reddish-yellow powder, almost insoluble in water, but soluble in nitric acid and in potassium hydroxide solution.

TESTS OF PURITY

Substances Insoluble in Acetic Acid. — To 2 gm. of lead oxide mixed with 5 c.c. of water add 10 c.c. of diluted acetic acid. No evolution of gas should take place. Now boil the liquid for several minutes, filter when cold, wash the undissolved residue, dry at 100° C., and weigh. The weight should not exceed 0.005 gm. (Indicating not more than 0.25 per cent.)

Carbonates. — 5 gm. of lead oxide, when heated even to the melting-point, should not lose more than 0.005 gm. in weight. (Indicating not more than 0.1 per cent CO_2.)

Copper, Iron, and Aluminum. — Dissolve 2 gm. of lead oxide in 10 c.c. of nitric acid and 5 c.c. of water, add to the clear solution 3 c.c. of concentrated sulphuric acid, and filter off the resulting precipitate. On adding to the filtrate an excess of ammonia water, the liquid should not acquire a blue color, nor should a precipitate form. (Indicating less than 0.001 per cent Cu, 0.002 per cent Fe, or 0.1 per cent Al.)

Nitrates and Nitrites. — Mix 1 gm. of lead oxide with 5 c.c. of water, and dissolve with the aid of 5 c.c. of diluted acetic acid. The blue color produced by adding 0.2 c.c. of 1 : 1000 indigo solution should not disappear on the addition of 10 c.c. of concentrated sulphuric acid and subsequent agitation. (Indicating less than 0.016 per cent as N_2O_5.)

Chlorides. — The solution of 1 gm. of lead oxide in 5 c.c. of nitric acid diluted with 20 c.c. of water should not become more than slightly opalescent upon the addition of silver nitrate solution. (Indicating less than 0.002 per cent Cl.)

Earths, Gypsum, and Alkalies. — Dissolve 1 gm. of lead oxide in 10 c.c. of diluted acetic acid, add 50 c.c. of water, and supersaturate with hydrogen sulphide gas. Filter off the precipitate, evaporate the filtrate, ignite the residue, and weigh. The weight should not exceed 0.003 gm. (Indicating not more than 0.3 per cent.)

LEAD SUBACETATE SOLUTION

A clear, colorless, liquid, of specific gravity 1.235 to 1.240. Lead subacetate solution is alkaline to litmus paper, but does not redden phenolphthalein.

TEST OF PURITY

Copper and Iron. — On acidifying 10 c.c. of lead subacetate solution with 2 c.c. of diluted acetic acid, and adding potassium ferrocyanide solution, a precipitate forms which should have a pure white color. (Indicating less than 0.0006 per cent Cu, or less than 0.0002 per cent Fe.)

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LIME, CHLORINATED

White cubes with chlorine-like odor, and liberating chlorine on the addition of hydrochloric acid. 100 parts treated with hydrochloric acid should afford at least 25 parts of Cl. Specially prepared in cube form for the generation of chlorine in the laboratory.

TEST OF PURITY

The value of this preparation depends entirely upon its active chlorine.

Quantitative Determination. — Mix 5 gm. of the powdered cubes with water, forming a thin paste. Wash this into a 500 c.c. graduated flask with water and dilute to the mark. To 50 c.c. of the thoroughly mixed material add a solution of 1 gm. of potassium iodide in 20 c.c. of water, followed by 5 c.c. of hydrochloric acid. Titrate the liberated iodine with decinormal sodium thiosulphate solution, of which at least 35.2 c.c. should be required.

1 c.c. of decinormal $Na_2S_2O_3 = 0.003546$ gm. of Cl, log. 54974.

LIME WATER

(Solution of Calcium Hydroxide)

A clear, colorless liquid having a strongly alkaline reaction.

TEST OF STRENGTH

Titrate 100 c.c. of lime water with normal solution of hydrochloric acid, using methyl orange as the indicator. Not less than 4 c.c., nor more than 4.5 c.c. of the normal solution of hydrochloric acid should be required to produce the red end-point.

1 c.c. of normal $HCl = 0.037053$ gm. of $Ca(OH)_2$, log. 56882.

LITMUS

A specially purified dye obtained from various species of lichens. It occurs in the form of friable lumps consisting chiefly of erythrolitmin and azolitmin.

A large number of formulas have been given for the preparation of litmus tincture, of which several are described by Glaser in his "Indikatoren der Azidimetrie und Alkalimetrie."

A very sensitive litmus tincture may be prepared as follows:

Dissolve 1 gm. of litmus in 100 c.c. of hot water, and add, drop by drop, dilute sulphuric acid until the liquid acquires a red color. Then boil for ten minutes in order to expel completely the carbon dioxide. Should the red color pass into blue during the boiling, the color is restored by again adding dilute sulphuric acid. Now add

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baryta water, drop by drop, until a violet color develops, set aside to deposit, and filter. Preserve the litmus tincture in bottles not completely filled, and simply cover with a loose cap, or stopper with a plug of cotton to keep out the dust.

TEST OF SENSITIVENESS

Add 0.2 c.c. of litmus tincture to 100 c.c. of distilled water, previously boiled for some time in a platinum dish to free it from carbon dioxide. The violet color of the liquid so obtained must change to red on the addition of 0.05 c.c. of decinormal solution of hydrochloric acid; or must pass into a pure blue color on the addition of 0.05 c.c. of decinormal solution of potassium hydroxide.

MAGNESITE

$MgCO_3$. Mol. Wt. 84.32

Hard pieces about the size of peas, soluble in hydrochloric acid with only a slight residue. It is used for the generation of carbon dioxide, especially as needed for the determination of nitrogen in organic substances.

TESTS OF PURITY

Loss on Ignition. — 1 gm. of magnesite, powdered and then dried at 100° C. before weighing, should leave a residue of about 0.5 gm. upon ignition to constant weight. (Indicating about 50 per cent.)

Quantitative Determination. — 1 gm. of magnesite is powdered and dissolved by the aid of heat in 50 c.c. of normal solution of sulphuric acid, and the solution is titrated with normal solution of potassium hydroxide, using methyl orange as indicator.

1 c.c. of normal $H_2SO_4 = 0.04216$ gm. of $MgCO_3$, log. 34242.

MAGNESIA MIXTURE

A solution prepared for the precipitation of phosphoric acid in quantitative determinations. It contains 110 gm. of crystallized magnesium chloride and 140 gm. of ammonium chloride in 1300 gm. of water and 700 gm. of ammonia water.

MAGNESIUM CARBONATE

(Magnesium Carbonate Basic)

White, light, loosely cohering, easily friable masses; or white, bulky powder. Basic magnesium carbonate is only very slightly soluble in water free from carbon dioxide, yet it imparts to it a slight alkaline reaction. In water impregnated with carbon dioxide

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and in aqueous solutions of ammonium salts, it is easily soluble. The composition of basic magnesium carbonate corresponds approximately to the formula $4MgCO_3 \cdot Mg(OH)_2 + 6H_2O$. Mol. Wt. 503.71. The content of magnesium is at least 24 per cent.

TESTS OF PURITY

Substances Soluble in Water. — Suspend 3 gm. of powdered basic magnesium carbonate in 90 c.c. of water, heat to boiling, and filter while hot. Evaporate 60 c.c. of the filtrate, and weigh the residue, which should not exceed 0.015 gm. (Indicating not more than 0.75 per cent.)

Sulphates and Substances Insoluble in Hydrochloric Acid. — 1 gm. of the basic magnesium carbonate should entirely dissolve in 5 c.c. of hydrochloric acid with 15 c.c. of water, and yield a colorless solution. (Indicating no insoluble substances present.) On adding barium chloride solution to this solution no change should occur. (Indicating less than 0.0125 per cent SO_3.)

Chlorides. — Dissolve 1 gm. of basic magnesium carbonate in 5 c.c. of nitric acid and 15 c.c. of water; on adding silver nitrate solution, not more than a slight opalescent turbidity should develop. (Indicating less than 0.002 per cent Cl.)

Barium. — The solution of 1 gm. of basic magnesium carbonate in 5 c.c. of hydrochloric acid and 15 c.c. of water should not be rendered turbid on the addition of diluted sulphuric acid. (Indicating less than 0.015 per cent Ba.)

Calcium and Alumina. — Dissolve 1 gm. of basic magnesium carbonate in 20 c.c. of diluted acetic acid and 30 c.c. of water. Boil, add 20 c.c. of ammonia water and some ammonium oxalate solution. The liquid should not become turbid within five minutes. (Indicating less than 0.06 per cent Ca and less than 0.6 per cent Al.)

Heavy Metals. — The solution of 1 gm. of basic magnesium carbonate in 20 c.c. of diluted acetic acid and 30 c.c. of water should be clear, and should show no change with hydrogen sulphide water. (Indicating none present.)

Iron. — The solution of 0.5 gm. of basic magnesium carbonate in 2 c.c. of hydrochloric acid and 20 c.c. of water should not acquire a blue color within one minute after the addition of 0.5 c.c. of potassium ferrocyanide solution. (Indicating less than 0.015 per cent Fe.)

Quantitative Determination. — On igniting 1 gm. of basic magnesium carbonate the residue should weigh at least 0.4 gm.

Dissolve 1 gm. of basic magnesium carbonate in 25 c.c. of normal hydrochloric acid solution, and titrate with normal potassium hydroxide solution, using methyl orange as indicator.

1 c.c. of normal HCl = 0.01216 gm. of Mg, log. 08493, or 0.02016 gm. of MgC log. 30449.

MAGNESIUM CHLORIDE

$MgCl_2 + 6H_2O$. Mol. Wt. 203.34

White, deliquescent, crystals soluble in about 0.6 part of cold, or in 0.3 part of hot, water, and in 5 parts of 85 per cent alcohol. The aqueous solution is neutral to litmus paper.

TESTS OF PURITY

Substances Insoluble in Alcohol. — 2 gm. of magnesium chloride should completely dissolve in 10 c.c. of 85 per cent alcohol, and yield a clear solution. (Indicating none present.)

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Sulphates. — The solution of 1 gm. of magnesium chloride in 20 c.c. of water should not be changed on the addition of 1 c.c. of hydrochloric acid and barium chloride solution. (Indicating less than 0.01 per cent SO_3.)

Phosphates and Arsenates. — Dissolve 3 gm. of magnesium chloride and 6 gm. of ammonium chloride in 24 c.c. of water, and add 12 c.c. of ammonia water. No turbidity or precipitate should form on standing fifteen hours. (Indicating less than 0.0016 per cent P_2O_5, and less than 0.05 per cent As_2O_5.)

Ammonium Salts. — On heating 3 gm. of magnesium chloride with 10 c.c. of sodium hydroxide solution (sp.gr. 1.3), no vapors of ammonia should be evolved (to be ascertained by moistened litmus paper). (Indicating less than 0.0011 per cent NH_3.)

Barium. — The solution of 1 gm. of magnesium chloride in 20 c.c. of water should afford no reaction on the addition of diluted sulphuric acid. (Indicating less than 0.002 per cent Ba.)

Heavy Metals and Calcium.— (*a*) The solution of 1 gm. of magnesium chloride in 20 c.c. of water must not be affected by hydrogen sulphide water. (Indicating no heavy metals present.)

(*b*) Dissolve 1 gm. of magnesium chloride in 20 c.c. of water, and add 1 gm. of ammonium chloride and 5 c.c. of ammonia water; no change should occur on the further addition of ammonium oxalate solution (indicating less than 0.005 per cent Ca), nor with a few drops of ammonium sulphide solution. (Indicating no heavy metals present.)

MAGNESIUM OXIDE

(Magnesia)

MgO. Mol. Wt. 40.32

I

MAGNESIUM OXIDE

A white, light, fine powder, almost insoluble in water. The preparation contains at least 88 per cent of magnesium oxide.

TESTS OF PURITY

Substances Soluble in Water. — Heat 3 gm. of magnesium oxide with 90 c.c. of water to boiling, and filter hot. The filtrate should have at most a slight alkaline reaction, and on evaporating 60 c.c. the weight of the residue should not exceed 0.015 gm. (Indicating not more than 0.75 per cent.)

Sulphates and Substances Insoluble in Hydrochloric Acid. — 1 gm. of magnesium oxide should completely dissolve in 10 c.c. of hydrochloric acid and 10 c.c. of water, yielding a clear solution. (Indicating no insoluble substances present.) This solution should not be rendered turbid within one minute after the addition of barium nitrate solution. (Indicating less than 0.375 per cent SO_3.)

Chlorides. — The solution of 0.5 gm. of magnesium oxide in 10 c.c. of nitric acid and 10 c.c. of water should not become more than opalescent on the addition of silver nitrate solution. (Indicating less than 0.010 per cent Cl.)

Carbonates. — Heat 0.5 gm. of magnesium oxide with 10 c.c. of water, and pour this mixture into 10 c.c. of dilute acetic acid. Solution should take place without any effervescence; only a slight evolution of gas may occur. (Indicating less than 4 per cent CO_2.)

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Nitrogen. — To the solution of 10 gm. of magnesium oxide in 100 c.c. of hydrochloric acid and 50 c.c. of water add 2 gm. of Devarda's metal and allow the mixture to stand, with frequent rotation, until the evolution of hydrogen has ceased. When the mixture is cold, add 100 c.c. of sodium hydroxide solution (N-free, sp.gr. 1.3) and distil off about 70 c.c., collecting the distillate in a receiver containing 2 to 3 c.c. of fifth-normal solution of hydrochloric acid and 10 c.c. of water. Titrate the distillate with fifth-normal solution of potassium hydroxide, using methyl orange as indicator. Not more than 0.2 c.c. of the fifth-normal solution of hydrochloric acid should be required to neutralize the ammonia obtained from the magnesium oxide. (Indicating not more than 0.0056 per cent N.)

Barium. — The solution of 1 gm. of magnesium oxide in 10 c.c. of hydrochloric acid and 10 c.c. of water should not be rendered turbid on the addition of diluted sulphuric acid. (Indicating less than 0.02 per cent Ba.)

Calcium. — On shaking 1 gm. of magnesium oxide with 20 c.c. of water and filtering, the filtrate, on the addition of ammonium oxalate solution, should not exhibit more than an opalescent turbidity within five minutes. (Indicating less than 0.01 per cent Ca.)

Heavy Metals. — The solution of 1 gm. of magnesium oxide in 20 c.c. diluted acetic acid and 30 c.c. of water should not be affected by hydrogen sulphide water. (Indicating none present.)

Iron. — The solution of 0.2 gm. of magnesium oxide in 2 c.c. of hydrochloric acid and 8 c.c. of water should not acquire a blue color within one minute after the addition of 0.5 c.c. of potassium ferrocyanide solution. (Indicating less than 0.025 per cent Fe.)

Quantitative Determination. — Dissolve 0.5 gm. of magnesium oxide in 30 c.c. of normal solution of hydrochloric acid and titrate with normal solution of potassium hydroxide, using methyl orange as indicator.

1 c.c. normal HCl = 0.02016 gm. MgO, log. 30449.

II

MAGNESIUM OXIDE, FREE FROM SULPHATES

TESTS OF PURITY

The tests as given for Magnesium Oxide should be made upon this article and in addition the following one:

Sulphates. — Dissolve 3 gm. of magnesium oxide in 20 c.c. of hydrochloric acid, dilute the solution to about 100 c.c., boil a few minutes, and add barium chloride solution. No precipitate of barium sulphate should form within fifteen hours. (Indicating less than 0.016 per cent SO_3.)

MAGNESIUM SULPHATE

$MgSO_4+7H_2O$. Mol. Wt. 246.50

Small, colorless, prismatic crystals, only very slightly efflorescent in air. Soluble in 1 part of cold, and in 0.3 part of boiling, water, but insoluble in alcohol. The aqueous solution is neutral to litmus paper.

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TESTS OF PURITY

Chlorides. — Dissolve 1 gm. of magnesium sulphate in 20 c.c. of water, and add 1 c.c. of nitric acid; the solution should not be affected by silver nitrate solution. (Indicating less than 0.0005 per cent Cl.)

Phosphates, Arsenates, and Heavy Metals. — The tests are to be carried out as detailed under Magnesium Chloride.

Iron. — 20 c.c. of the 1 : 20 aqueous solution, slightly acidified with nitric acid and boiled, should not show a red color on the addition of potassium sulphocyanate solution. (Indicating less than 0.0008 per cent Fe.)

Arsenic. — A mixture of 1 gm. of powdered magnesium sulphate and 3 c.c. of stannous chloride solution should not acquire a dark color on standing one hour. (Indicating less than 0.0015 per cent As.)

MAGNESIUM AND AMMONIUM CHLORIDE

$MgCl_2NH_4Cl+6H_2O$. Mol. Wt. 256.84

Rhombic crystals, or white, crystalline powder, easily soluble in water. Its principal use is in the preparation of magnesia mixture for the determination of phosphoric acid.

TESTS OF PURITY

Sulphates; Heavy Metals; Calcium and Barium. — The tests are to be made as detailed under Magnesium Chloride.

Phosphates and Arsenates. — Dissolve 5 gm. of magnesium and ammonium chloride in 10 c.c. of water, and add 5 c.c. of ammonia water. No turbidity or precipitate should form within fifteen hours. (Indicating less than 0.001 per cent P_2O_5, and less than 0.005 per cent As_2O_5.)

MANGANESE CHLORIDE

(Manganous Chloride)

$MnCl_2+4H_2O$. Mol. Wt. 197.91

Reddish, hygroscopic crystals, easily soluble in water (about 1 : 1) and also soluble in alcohol.

TESTS OF PURITY

Sulphates. — Dissolve 1 gm. of manganous chloride in 10 c.c. of water, add 1 c.c. of hydrochloric acid and some barium chloride solution. No change should be noticeable. (Indicating less than 0.01 per cent SO_3.)

Chlorine. — The solution of 1 gm. of manganous chloride in 40 c.c. of water and 5 c.c. of 16 per cent sulphuric acid should not be rendered blue within one minute by zinc iodide-starch solution. (Indicating less than 0.0023 per cent Cl.)

Calcium. — 20 c.c. of the 1 : 20 aqueous solution should not be rendered turbid

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within one minute after the addition of 2 c.c. of ammonium oxalate solution.* (Indicating less than 0.03 per cent Ca.)

Iron. — Dissolve 1 gm. of manganous chloride in 15 c.c. of water, add 1 c.c. of hydrochloric acid, 3 c.c. of chlorine water, and heat to boiling. The solution, when cold, should not acquire a red color on the addition of potassium sulphocyanate solution. (Indicating less than 0.0008 per cent Fe.)

Salts of Magnesium and the Alkalies. — Precipitate the manganese from a solution of 3 gm. of manganous chloride in 50 c.c. of water by adding ammonium carbonate solution (5 gm. of ammonium carbonate in 50 c.c. of water). Filter, evaporate the filtrate, and ignite the residue. It should not weigh more than 0.001 gm. (Indicating not more than 0.033 per cent.)

Zinc. — Dissolve 1 gm. of manganous chloride and 1 gm. of sodium acetate in 10 c.c. of water, and add a few drops of acetic acid and hydrogen sulphide water. No white precipitate soluble in hydrochloric acid should form. (Indicating less than 0.2 per cent Zn.)

Other Foreign Metals. — Dissolve 1 gm. of manganese chloride in 20 c.c. of water and add 5 c.c. of hydrochloric acid. This solution should not be affected by hydrogen sulphide water. (Indicating none present.)

MANGANESE DIOXIDE

(Manganese Per- or Super-Oxide; Pyrolusite)

MnO_2. Mol. Wt. 86.93

Grayish-black pieces, soluble in cold, concentrated hydrochloric acid forming manganese tetrachloride, which on heating decomposes into manganous chloride and chlorine. In hot, concentrated sulphuric acid manganese dioxide dissolves with the evolution of oxygen.

TEST OF PURITY

Quantitative Determination.† — Weigh off 1.0866 gm. of the manganese dioxide, previously very finely powdered and dried to constant weight at 100° C. Place in a flask provided with a Bunsen rubber valve, and add 75 c.c. (3 pipettefuls of 25 c.c. each) of a solution of 100 gm. of pure, crystallized ferrous sulphate and 100 c.c. of pure, concentrated sulphuric acid in 1000 c.c. of water.‡ Close the flask with the stopper bearing the Bunsen valve, and heat until the manganese dioxide is completely decomposed, leaving no dark-colored residue. Then cool, making sure the valve is closed as indicated by the collapsing of the rubber tubing.

When cold, dilute with 200 c.c. of water, and titrate with seminormal potassium permanganate solution until the faint red color no longer disappears, but persists for half a minute (further decoloration is neglected). From the quantity of permanganate solution corresponding by calculation to the 75 c.c. of ferrous sulphate solution deduct now the quantity of permanganate solution actually used. Each cubic centimeter of the difference represents 0.02173 gm., or 2 per cent, of MnO_2.

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* On allowing this mixture to stand for some time, manganous oxalate separates in the form of acicular crystals.

† Lunge, Chem.-Tech. Untersuch.-Meth., 5 ed., I, 486.

‡ This ferrous sulphate solution is standardized against seminormal potassium permanganate solution on the same day, using the same pipette.

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MANGANESE METAPHOSPHATE SOLUTION

A reagent for the detection of albumin, prepared as directed by L. Blum.*

The solution should have a pink color. If in the course of time it becomes colorless, it is no longer of use as a reagent for albumin.

MANGANESE SULPHATE

(Manganous Sulphate)

$MnSO_4+4H_2O$. Mol. Wt. 223.06.

Pale-red, monoclinic crystals, soluble in 0.8 part of water, and insoluble in alcohol. The aqueous solution is neutral to litmus paper.

TESTS OF PURITY

Chlorides. — The solution of 1 gm. of manganese sulphate in 20 c.c. of water should not be rendered more than slightly opalescent on the addition of 1 c.c. of nitric acid, followed by silver nitrate solution. (Indicating less than 0.002 per cent Cl.)

Substances Reducing Potassium Permanganate. — Dissolve 15 gm. of manganese sulphate in 200 c.c. of water with 3 c.c. each of phosphoric acid (sp.gr. 1.7) and concentrated sulphuric acid. Not more than 0.1 c.c. of decinormal potassium permanganate solution should be necessary to impart a slight red color to the solution. (Indicating at most a trace present)

Calcium, Iron, Zinc, Other Foreign Metals, and Salts of Magnesium and the Alkalies. — The tests are carried out as detailed under Manganese Chloride.

MERCURY

Hg. Atomic Wt. 200.6

A liquid, silver-white metal, which solidifies at about −39° C., and boils at about 357° C. Its specific gravity is 13.56.

TESTS OF PURITY

Foreign Metals. — (*a*) Mercury should have a bright surface which should not be affected by shaking with pure air. (Indicating none present.)

(*b*) On heating 20 gm. of mercury in a porcelain dish under a good draft, no weighable residue should remain. (Indicating less than 0.0025 per cent.)

(*c*) On boiling 5 gm. of mercury with 5 c.c. of water and 4.5 gm. of sodium thiosulphate for about one minute, in a test-tube, the mercury must not lose its luster, and should acquire at most only a slight yellowish tinge. (Indicating at most a trace present.)

(*d*) Mercury should dissolve completely in nitric acid. (Indicating no insoluble metals present.)

* L. Blum, Chem. Ztg., **11** (Rep.), 24 (1887); *abst.*, J. Chem. Soc., **52**, 1003 (1887).

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MERCURY BICHLORIDE

(Mercuric Chloride; Corrosive Sublimate)

$HgCl_2$. Mol. Wt. 271.52

White, translucent, prismatic crystals. Mercuric chloride is soluble in 16 parts of cold, and in 3 parts of boiling, water; in 3 parts of alcohol (about 85 per cent by weight), and in about 17 parts of ether* (sp.gr. 0.720). The aqueous solution is acid to litmus paper; addition of sufficient sodium chloride effects neutrality.

TESTS OF PURITY

Foreign Matter not Precipitated by Hydrogen Sulphide. — Dissolve 5 gm. of mercuric chloride in 100 c.c. of water, add 5 c.c. of hydrochloric acid, and pass into the solution hydrogen sulphide gas until the mercury has been completely precipitated. Then filter, evaporate the filtrate to dryness on the water-bath, and ignite gently. No weighable residue should remain. (Indicating less than 0.01 per cent.)

Arsenic. — Shake the mercury sulphide obtained in the above test with a mixture of 5 c.c. of ammonia water and 45 c.c. of water, filter, and acidulate the filtrate with hydrochloric acid. Neither a yellow color nor a yellow precipitate should appear. (Indicating less than 0.008 per cent As.)

Mercurous Chloride and Other Substances Insoluble in Ether. — 1 gm. of powdered mercuric chloride should be completely soluble in 25 c.c. of ether. (Indicating none present.)

Quantitative Determination. — Dissolve 1 gm. of mercuric chloride in 100 c.c. of water and titrate† the solution with semi-normal solution of potassium cyanide, using phenolphthalein as indicator.

1 c.c. of seminormal KCN = 0.06788 gm. $HgCl_2$, log. 83174.

MERCURY NITRATE

(Mercurous Nitrate)

$HgNO_3+H_2O$. Mol. Wt. 280.63

Colorless, monoclinic tables or prisms, soluble in about 2 parts of warm water, affording a solution with an acid reaction. On the addition of much water the solution is decomposed with the separation of a light-yellow precipitate. Solution is best effected by means of water acidified with nitric acid.

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* The extent of solubility of mercuric chloride in ether depends upon the alcohol content of the ether.

† E. Rupp, Apotheker-Zeitung, 24, 939 (1909); *abst.*, C. A., 4, 1084 (1910).

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TESTS OF PURITY

Non-volatile Matter. — On igniting 2 gm. of mercurous nitrate, no weighable residue should remain. (Indicating less than 0.025 per cent.)

Mercuric Salts. — Dissolve 1 gm. of mercurous nitrate in 5 c.c. of water with 3 to 5 drops of nitric acid. Dilute the solution with 15 c.c. of water, add 1 c.c. of hydrochloric acid, filter, and add hydrogen sulphide water to the filtrate. More than traces of a precipitate should not be visible. (Indicating at most a trace present.)

Quantitative Determination. — Dissolve 1 gm. of mercurous nitrate in 25 c.c. of nitric acid in a 100 c.c. graduated flask, add decinormal potassium permanganate solution until the mixture acquires a red color lasting several minutes, or until the separation of brown flocks, and then add powdered ferrous sulphate until the solution is clear and decolorized. Fill to the mark with water, mix, and titrate of 20 c.c. of the solution with decinormal ammonium sulphocyanate solution, after the addition of 5 c.c. of a cold saturated solution of ferric-ammonium sulphate.

1 c.c. of decinormal $NH_4CNS = 0.01403125$ gm. $HgNO_3 + H_2O$, log. 14710.

MERCURY OXIDE RED

(Mercuric Oxide Red)

HgO. Mol. Wt. 216.6

A yellowish-red, crystalline powder, almost insoluble in water, easily soluble in diluted nitric acid, forming a clear solution, and in diluted hydrochloric acid, forming a solution at most slightly turbid.

TESTS OF PURITY

Non-volatile Matter. — On igniting 2 gm. of mercuric oxide, no weighable residue should remain. (Indicating less than 0.025 per cent.)

Chlorides. — The solution of 1 gm. of mercuric oxide in 5 c.c. of nitric acid and 15 c.c. of water should not be rendered more than slightly opalescent on adding silver nitrate solution. (Indicating less than 0.002 per cent Cl.)

Sulphates. — The solution of 1 gm. of mercuric oxide in 5 c.c. of nitric acid and 15 c.c. of water should show no change on the addition of barium nitrate solution. (Indicating less than 0.175 per cent SO_3.)

Nitrates. — Dissolve 1 gm. of mercuric oxide in 2 c.c. of water and 2 c.c. of concentrated sulphuric acid, and overlay this mixture, when cold, with ferrous sulphate solution. No colored zone should form at the plane of contact of the two layers. (Indicating less than 0.016 per cent N_2O_5.)

Quantitative Determination. — Dissolve 1 gm. of the mercuric oxide in 25 c.c. of nitric acid in a 100 c.c. graduated flask, and dilute with water to the mark. Titrate 20 c.c. of this solution with decinormal solution of ammonium sulphocyanate, using 5 c.c. of cold, saturated solution of ferric-ammonium sulphate as indicator.

1 c.c. of decinormal $NH_4CNS = 0.01083$ gm. of HgO, log. 03463.

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MERCURY OXIDE YELLOW

(Mercuric Oxide Yellow)

HgO. Mol. Wt. 216.6

A yellow, amorphous powder, almost insoluble in water, but easily soluble in diluted nitric or hydrochloric acid. On being shaken with oxalic acid solution, yellow mercuric oxide changes gradually into a white crystalline powder.

TESTS OF PURITY

The tests for non-volatile matter, chlorides, sulphates, and nitrates, and the quantitative determination are to be carried out as detailed under Mercury Oxide Red.

MERCURY AND POTASSIUM IODIDE

(Mercuric Potassium Iodide)

$HgI_2 2KI$. Mol. Wt. 786.48

Sulphur-yellow, crystalline pieces, deliquescent in moist air.

TEST OF PURITY

Solubility. — 5 gm. of mercuric potassium iodide should completely dissolve in 10 c.c. of water, and yield a clear solution which should remain clear even after adding 80 c.c. of water.

METAPHENYLENEDIAMINE HYDROCHLORIDE

(Metadiaminobenzene Hydrochloride)

$C_6H_4(NH_2)_2 2HCl$. Mol. Wt. 181.02

A white, or slightly reddish-white, crystalline powder, easily soluble in water. Metaphenylenediamine hydrochloride is used as a reagent in the form of a 1 : 200 solution. Should this solution have a color, it is to be decolorized before using by heating with previously ignited animal charcoal.

TESTS OF PURITY

Inorganic Impurities. — On igniting 1 gm. of metaphenylenediamine hydrochloride no weighable residue should remain. (Indicating less than 0.05 per cent.)

Quantitative Determination. — Dissolve 0.5 gm. of metaphenylenediamine hydrochloride in 50 c.c. of 85 per cent alcohol in a glass-stoppered flask, and titrate this solution with normal potassium hydroxide solution, using Poirrier's blue as indicator.

1 c.c. of normal KOH = 0.09051 gm. of $C_6H_4(NH_2)_2 \cdot 2HCl$, log. 95670.

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METHYL ORANGE

(Sodium Salt of Paradimethylaminoazobenzenesulphonic Acid)

$(CH_3)_2NC_6H_4N:NC_6H_4SO_3Na$. Mol. Wt. 327.21

An orange-yellow powder, easily soluble in water. As an indicator, a solution of 0.1 gm. in 100 c.c. of water is used.

TEST OF SENSITIVENESS

To 100 c.c. of distilled water in a Jena flask add 1 drop of the above methyl orange solution. On adding to this solution 0.05 c.c. of decinormal solution of hydrochloric acid, the light yellow color of the water should change to a red, and on the further addition of 0.05 c.c. of decinormal solution of potassium hydroxide the original color should be restored.

METHYL RED

(Methyl Red Indicator)

$(CH_3)_2NC_6H_4N:NC_6H_4COOH$. Mol. Wt. 269.15

Glistening, violet crystals, almost insoluble in water, soluble in alcohol and in glacial acetic acid. As an indicator, a solution of 0.2 gm. in 100 c.c. of alcohol is used.

TEST OF SENSITIVENESS

Mix 1 or 2 drops of methyl red solution with 100 c.c. of water which has been freed from carbon dioxide by long boiling in a platinum dish. The yellowish-red color of the mixture should be changed to yellow by the addition of 0.05 c.c. of centinormal potassium hydroxide solution, and on further adding 0.1 c.c. of centinormal hydrochloric acid solution the fluid should become red.

NOTE. — Regarding the use of methyl red as an indicator, see E. Rupp and R. Loose, Ber., **41**, 3905 (1908); *abst.*, J. Chem. Soc., **96**, II, 90 (1909). Tizard, J. Chem. Soc., **97**, II, 2477 (1910).

NITROBENZALDEHYDE, ORTHO-

$C_6H_4(NO_2)CHO$. Mol. Wt. 151.05

Light-yellow needles, melting between 45° and 46° C., and easily soluble in alcohol and in ether.

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NITRON
FOR THE GRAVIMETRIC DETERMINATION OF NITRIC ACID
(1.4 Diphenyl - 3.5 Endanilodihydrotriazol)

$C_{20}H_{16}N_4$. Mol. Wt. 312.17

Shining, yellow leaflets, or amorphous powder, melting at 189° C. with decomposition. It is soluble in alcohol, chloroform, acetone, and ethyl acetate, difficultly soluble in ether, and insoluble in water. In alcohol, the substance undergoes partial decomposition, indicated by the red color of the solution. Nitron is used as a reagent in the form of a 10 per cent solution in 5 per cent acetic acid, according to the method of M. Busch. This solution should be kept in well-stoppered bottles and protected from light.

TEST OF PURITY

Solubility.—1 gm. of nitron should easily and almost completely dissolve in 10 c.c. of 5 per cent acetic acid.

NOTE. — Literature: M. Busch, "Gravimetric Determination of Nitric Acid", Ber., **38**, 861 (1905); *abst.*, J. Chem. Soc., **88**, II, 282 (1905). C. Paal and E. Weidenkaff, "Determination of Nitric Acid in Organic Salts," Ber., **38**, 1688 (1905); *abst.*, J. Chem. Soc., **88**, I, 436 (1905). M. Busch, "Determination of Nitric Acid in Water," Z. Nahr. Genussm., **9**, 464 (1905); *abst.*, J. Chem. Soc., **90**, II, 282 (1905). A. Gutbier, "Gravimetric Determination of Nitric Acid by Means of Nitron acc. to M. Busch" Z. angew. Chem., **18**, 494 (1905); *abst.*, J. Chem. Soc., **88**, II, 418 (1905). G. Lunge and E. Berl, "Analysis of Mixed Acids (Sulphuric and Nitric Acids)", Z. angew. Chem., **18**, 1681 (1905); *abst.*, J. Chem. Soc., **90**, II, 49 (1906). M. Busch, "Determination of Nitrate in the Presence of Nitrite," Ber., **39**, 1401 (1906); *abst.*, J. Chem. Soc., **90**, II, 392 (1906). Compare W. Traube and A. Biltz, Ber., **39**, 168 (1906); *abst.*, J. Chem. Soc., **90**, II, 159 (1906). M. Busch, "A New Method for the Determination of the Nitrogen-content of Nitrocellulose," Z. angew. Chem., **30**, 1329 (1906) [or Z. ges. Schiess-Sprengstoffw., **1**, 232 (1906)]; *abst.*, J. Chem. Soc., **90**, II, 708 (1906). C. Paal and G. Mehrtens, "Gravimetric Determination of Saltpeter in Meat," Z. Nahr. Genussm., **12**, 410 (1906); *abst.*, J. Chem. Soc., **90**, II, 898 (1906). H. L. Visser, "The Use of Nitron in Microchemical Analysis," Chem. Weekblad, **3**, 743 (1906); *abst.*, Chem. Zentr., (5) **11**, 302 (1907); *abst.*, J. Chem. Soc., **92**, II, 394 (1907). J. Litzendorff, "The Use of Nitron for the Determination of Nitric Acid in Soil and in Plants," Z. angew. Chem., **20**, 2209 (1907); *abst.*, J. Chem. Soc., **94**, II, 130 (1908). M. Busch and G. Blume, "Quantitative Determination of Picric Acid," Z. angew. Chem., **21**, 354 (1908); *abst.*, J. Chem. Soc. **94**, II, 328 (1908). H. Franzen and E. Löhmann, "The Use of Nitron in the Determination of Nitric Acid in Fluids Containing Much Organic Matter," J. prakt. Chem., **79**, 330 (1909); *abst.*, J. Chem. Soc., **96**, II, 517 (1909). A. Hes, "Gravimetric Determination of Nitric Acid," Z. anal. Chem., **48**, 81 (1909); *abst.*, J. Chem. Soc., **96**, II, 265 (1909). M. Busch, "Reply to A. Hes" (loc. cit.), Z. anal. Chem., **48**, 368 (1909); *abst.*, J. Chem. Soc., **96**, II, 615 (1909). P. Pooth, "A Contribution to the Gravimetric Determination of Nitric Acid acc. to M. Busch by Means of Nitron," Z. anal. Chem., **48**, 365 (1909); *abst.*,

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J. Chem. Soc., **96**, II, 615 (1909). C. Paal and A. Ganghofer, "The Determination of Nitric Acid with Nitron," Z. anal. Chem., **48**, 545 (1909); *abst.*, J. Chem. Soc., **96**, II, 759 (1909). C. Paal and A. Ganghofer, "Determination of Saltpeter in Meat with Nitron," Z. Nahr. Genussm., **19**, 322 (1910); *abst.*, J. Chem. Soc., **98**, II, 453 (1910). L. Radlberger, "The Use of M. Busch's Nitron for the Analysis of Chili Saltpeter," Oesterr. Ung. Z. Zuckerind., **39**, 433; *abst.*, Chem. Zentr., (5) **14**, II, 685 (1910); C. A., **5**, 1721 (1911).

NITROPHENOL, ORTHO-

$C_6H_4OH\,NO_2$. Mol. Wt. 139.05

Sulphur-yellow needles or prisms, melting at 44° to 45° C., easily soluble in alcohol and in ether, freely soluble in hot water, and but slightly soluble in cold water.

NITROPHENOL, PARA-

$C_6H_4OH\,NO_2$. Mol. Wt. 139.05

Colorless needles or monoclinic prisms, melting at 112° C. and easily soluble in alcohol; to some extent also in water.

NITROSOBETANAPHTHOL

$C_{10}H_6(NO)(OH)$. Mol. Wt. 173.07

Orange-brown crystals, melting at 109° C., insoluble in cold water, very difficultly soluble in boiling water, and very easily soluble in ether, benzene, and hot alcohol. It is used principally for the qualitative separation of nickel and cobalt.

PALLADIUM

Pd. Atomic Wt. 106.7

Palladium occurs in the form of foil and wire which in appearance greatly resemble platinum; also in the form of palladium-sponge, a gray spongy mass, and as palladium black. The compact metal is soluble in hot nitric and in nitrohydrochloric acids. Palladium sponge is soluble also in hydrochloric acid in the presence of air.

TESTS OF PURITY

Differentiation Between Palladium Foil and Platinum Foil. — On placing 1 drop of an alcoholic solution of iodine on palladium foil, and allowing it to

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evaporate spontaneously in the air, a black spot will be formed on the palladium which will disappear on heating the foil to redness. On platinum foil similarly treated, no spot is formed.

Copper and Iron. — Dissolve the palladium in nitrohydrochloric acid, and evaporate the excess of acid on the water-bath. Dissolve the residue in water, and add ammonia water until the flesh-colored precipitate of ammonium palladous chloride first formed redissolves. Then pass into the solution gaseous hydrochloric acid, whereby the palladium is precipitated as yellow palladosamine chloride, while iron and copper remain in the solution. The precipitate is filtered off, and the filtrate treated with ammonia water in excess. No coloration or precipitation should ensue. (Indicating none present.)

NOTE. — Regarding the quantitative determination of palladium and its separation from other metals, see Fresenius, Anleitung zur quantitativen chemishen Analyse, 6 ed., **1**, 348, 481; Fresenius-Cohn, Quantitative Chemical Analysis, **1**, 389. H. Erdman and O. Makowka, Ber., **37**, 2694 (1904); *abst.*, J. Chem. Soc., **86**, II, 594 (1904). Z. anal. Chem., **46**, 141, 146 (1907); *abst.*, J. Chem. Soc., **92**, 403 (1907). A. Gutbier and F. Falco, Z. anal. Chem., **48**, 555 (1909); *abst.*, J. Chem. Soc., **96**, II, 768 (1909). Z. anal. Chem., **49**, 287, 492 (1910); *abst.*, J. Chem. Soc., **98**, II, 459, 756 (1910).

PALLADIUM CHLORIDE

(Palladous Chloride)

$PdCl_2$. Mol. Wt. 177.62

A dark-brown powder, yielding a turbid solution in water in consequence of the formation of a basic salt; clearly soluble on adding hydrochloric acid. The preparation contains about 60 per cent of palladium, the theoretical per cent calculated from the formula $PdCl_2$ being 60.08.

TEST OF PURITY

Quantitative Determination of Palladium. — Dissolve 0.5 gm. of palladium chloride in 2 to 3 c.c. of hydrochloric acid, dilute with 100 c.c. of water, and completely precipitate the palladium by passing into the solution acetylene gas. Filter off the precipitate, wash it free from chlorides with water, ignite it, still moist, in a porcelain crucible, cautiously at first, then more strongly, finally in a current of hydrogen, and weigh.

PALLADIUM NITRATE

(Palladous Nitrate)

$Pd(NO_3)_2$. Mol. Wt. 230.72

A brown, deliquescent salt, yielding a turbid solution with water, due to the almost invariable presence of some basic salt.

TEST OF PURITY

Quantitative Determination of Palladium. — Dissolve 0.5 gm. of palladium nitrate in 5 c.c. of nitric acid and 10 c.c. of hydrochloric acid, heating on the

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water-bath. Evaporate the solution, dissolve the residue in 10 c.c. of hydrochloric acid, and again evaporate to dryness. Dissolve the residue in 2 to 3 c.c. of hydrochloric acid, dilute with 100 c.c. of water, and conduct acetylene gas into the solution until the palladium is completely precipitated. Filter off the precipitate, wash it with water until free from chlorides, ignite it, still moist, in a porcelain crucible, cautiously at first, then more strongly, finally in a current of hydrogen, and weigh.

PALLADIUM AND SODIUM CHLORIDE

(Sodium Palladous Chloride)

$PdCl_2 2NaCl + 3H_2O$. Mol. Wt. 348.59

A reddish-brown powder, soluble in water.

TEST OF PURITY

Quantitative Determination of Palladium.—This test is to be carried out as described under Palladium Chloride.

PHENACETOLIN

A brown dye, soluble in alcohol. Phenacetolin is a product resulting from the action of concentrated sulphuric acid on glacial acetic acid and phenol. The indicator solution is prepared by digesting 1 gm. of phenacetolin with warm alcohol, then diluting to 100 c.c., and filtering.

TEST OF SENSITIVENESS

Add 2 drops of the indicator solution to 100 c.c. of distilled water. On the addition of 0.05 c.c. of decinormal potassium hydroxide solution, the light-brown color of the liquid should pass into pink; on the further addition of 0.05 c.c. of decinormal hydrochloric acid, the color should change to a golden yellow.

NOTE. — Concerning the properties and use of phenacetolin as an indicator, see Glaser, Indikatoren der Azidimetrie und Alkalimetrie, 69. See also Cohn, Indicators and Test Papers, 2 ed., page 122 (1902).

PHENOLPHTHALEIN

$C_{20}H_{14}O$. Mol. Wt. 318.11

A white, or yellowish-white, powder easily soluble in alcohol, and melting at about 260° C. Phenolphthalein is used as an indicator in the form of a solution of 1 gm. in 100 c.c. of neutral 95 per cent alcohol.

TESTS OF PURITY

Non-volatile Matter. — 0.5 gm. of phenolphthalein when ignited should yield no weighable residue. (Indicating less than 0.1 per cent.)

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Solubility in Alcohol. — 1 gm. of phenolphthalein should give a clear solution with 15 c.c. of 95 per cent alcohol. The 1 : 100 alcoholic solution should be colorless.

Sensitiveness. — To 250 c.c. of water, which has been well boiled and then cooled, add 3 to 5 drops of phenolphthalein solution; the solution should require not more than 0.05 c.c. of decinormal potassium hydroxide solution to produce a coloration.

Fluorane. — 0.5 gm. of phenolphthalein should completely dissolve in a mixture of 1 c.c. of sodium hydroxide solution (sp.gr. 1.168) and 50 c.c. of water. (Indicating none present.)

PHENYLHYDRAZINE

$C_6H_5NHNH_2$. Mol. Wt. 108.08

A colorless, or slightly yellowish, highly refractive liquid, boiling at 243° C., and solidifying when cold with the formation of monoclinic plates, melting at 19° C. Phenylhydrazine is but slightly soluble in cold water, but is more readily soluble in hot water, and is easily soluble in alcohol and in ether. It is very sensitive to air.

TEST OF PURITY

Solubility. — 2 c.c. of phenylhydrazine, when shaken with 20 c.c. of 5 per cent acetic acid, should afford a clear solution.

PHLOROGLUCIN

$C_6H_3(OH)_3+2H_2O$. Mol. Wt. 162.08

A white, or slightly yellowish, crystalline powder, which loses its water of crystallization at 100° C., and which, on being rapidly heated, melts at 217° to 219° C., but which, when slowly heated, melts at a much lower temperature (200° to 209° C.).* Phloroglucin is soluble in about 95 parts water, and in 10 parts of alcohol (about 85 per cent by weight.)

TEST OF PURITY

Diresorcin. — Heat to boiling a solution of 0.1 gm. of phloroglucin in 10 c.c. of acetic anhydride, cool the solution, and superimpose it upon 10 c.c. of concentrated sulphuric acid. No violet zone should appear at the contact planes of the liquids. (Indicating none present.)

* A. Baeyer, Ber., **19**, 2186 (1886); *abst.*, J. Chem. Soc., **50**, 1020 (1886).

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PLATINUM

Pt. Atomic Wt. 195.2

Platinum occurs in compact form as foil or wire, and in a state of very fine subdivision as platinum sponge and platinum black. Compact platinum has a silver-white color; platinum sponge is a grayish, spongy mass, while platinum black is a black powder.

TESTS OF PURITY

Foreign Metals. — Dissolve 1 gm. of the metal in nitrohydrochloric acid, evaporate the solution to dryness on the water-bath, and convert the residual platinum chloride into platinum sponge by strongly igniting. On now treating the sponge with nitric acid and filtering, the filtrate should not leave more than traces of residue on being evaporated. (Indicating at most a trace present.)

Silver. — Dissolve platinum in nitrohydrochloric acid, evaporate the solution to dryness on the water-bath, and dissolve the residue in water. No white residue should remain. (Indicating none present.)

NOTE.—Regarding the further testing of platinum for slight impurities, see the paper by F. Mylius and P. Foerster: The Preparation and Examination of Pure Platinum, Ber., **25**, 665 (1892); *abst.*, J. Chem. Soc., **62**, 789 (1892).

PLATINUM CHLORIDE

(Platinic Chloride; Chlorplatinic Acid)

$H_2PtCl_6+6H_2O$. Mol. Wt. 518.07

A brownish-red, crystalline, very hygroscopic, saline mass, soluble in water, alcohol, and ether, with yellow color. The aqueous solution is acid to litmus paper.

TESTS OF PURITY

Solubility in Absolute Alcohol. — 1 gm. of platinum chloride should dissolve completely in 10 c.c. of absolute alcohol, yielding a clear solution. Platinic chloride should also afford a clear, pure yellow solution with water; any red or dark-brown tinge would indicate the presence of platinous chloride or iridium.

Metals Soluble in Nitric Acid. — Strongly ignite 2 gm. of platinic chloride. A residue of platinum sponge weighing 0.752 gm. should be obtained. Digest this residue with dilute nitric acid (5 c.c. of nitric acid and 20 c.c. of water) on the water-bath for fifteen minutes, then filter, evaporate the filtrate on the water-bath, and ignite the residue so obtained. The weight of the latter should not exceed 0.005 gm. (Indicating not more than 0.25 per cent.)

Sulphates. — The solution of 1 gm. of platinic chloride in 20 c.c. of water should give no precipitate of barium sulphate on adding barium chloride solution, and setting aside three hours. (Indicating less than 0.0075 per cent SO_3.)

Nitrates. — On mixing 2 c.c. of the 1 : 10 solution with 2 c.c. of concentrated sulphuric acid, and overlaying this mixture with 2 c.c. of ferrous sulphate solution, no brownish-red zone should develop at the contact planes of the two liquids. (Indicating less than 0.08 per cent N_2O_5.)

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Barium Salts. — The 1 : 20 solution of platinic chloride should afford no precipitate of barium sulphate on adding a few drops of sulphuric acid, and allowing to stand three hours. (Indicating less than 0.002 per cent Ba.)

POIRRIER'S BLUE

(Poirrer's Blue C_4B, Indicator)

The ammonium salt of triphenylrosanilinetrisulphonic acid.

A dark-blue powder, soluble in water and in alcohol. A solution of 0.2 gm. in 100 c.c. of water is used as an indicator.*

TEST OF SENSITIVENESS

To 45 c.c. of absolute alcohol, contained in a glass-stoppered flask of 50 c.c. capacity, add 3 to 5 drops of the above solution of Poirrier's blue and then by drops decinormal solution of potassium hydroxide, shaking vigorously after each drop, until the blue color has changed into red. On now adding 0.05 c.c. of decinormal solution of hydrochloric acid, the red color should change to blue; on further addition of 0.05 c.c. of decinormal potassium hydroxide solution the red color should return.

POTASSIUM ACETATE SOLUTION

A clear, colorless liquid, neutral, or at most slightly acid to phenolphthalein; specific gravity 1.176 to 1.180. 100 parts contain about 33 parts of potassium acetate.

TESTS OF PURITY

Chlorides. — Dilute 5 c.c. of potassium acetate solution with 20 c.c. of water, and add 5 c.c. of nitric acid followed by silver nitrate solution. At most a slight opalescent turbidity should develop. (Indicating less than 0.0005 per cent Cl.)

Sulphates. — Dilute 10 c.c. of potassium acetate solution with 10 c.c. of water, acidify with hydrochloric acid, and add barium chloride solution. No change should appear. (Indicating less than 0.01 per cent SO_3.)

Heavy Metals. — (*a*) 15 c.c. of potassium acetate solution diluted with 15 c.c. of water should not become colored on the addition of hydrogen sulphide water; nor should a precipitate form. On now adding a few drops of ammonium sulphide solution, neither a green or brown color nor a precipitate should appear. (Indicating none present.)

(*b*) On diluting 5 c.c. of potassium acetate solution with 20 c.c. of water and adding 1 c.c. of hydrochloric acid, the solution should not acquire a red color with potassium sulphocyanate solution. (Indicating less than 0.00015 per cent Fe.)

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* Poirrier's blue is very sensitive to acids and consequently is very well adapted to the titration of salts of weak bases, for example, alkaloidal salts, which in alcoholic solution react toward the indicator like free acids. R. Engel, Compt. rend., **102**, 214, 262 (1886); *abst.*, J. Soc. Chem. Ind., **5**, 392 (1886). J. Messner, Z. angew. Chem., **16**, 469 (1903); *abst.*, J. Chem. Soc., **84**, 519 (1903). See also Z. anal. Chem., **27**, 38 (1888).

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Calcium. — Dilute 10 c.c. of potassium acetate solution with 10 c.c. of water, and add ammonium oxalate solution. No precipitate of calcium oxalate should form on standing three hours. (Indicating less than 0.002 per cent Ca.)

POTASSIUM ANTIMONATE

(Potassium Acid Pyroantimonate)*

$K_2H_2Sb_2O_7+4H_2O$. Mol. Wt. 504.58

A white, granular, crystalline powder, difficultly soluble in cold water, and more readily soluble in boiling water.

TESTS OF PURITY

Suitability as a Reagent for Sodium.†—The aqueous solution of 1 gm. of potassium antimonate in 100 c.c. of boiling water should be neutral to litmus paper.

On adding 1 gm. of potassium chloride, dissolved in 10 c.c. of water, to 20 c.c. of the 1 : 100 aqueous solution, no precipitate should form within fifteen minutes.

To 20 c.c. of the 1 : 100 aqueous solution add 1 gm. of ammonium chloride dissolved in 10 c.c. of water containing 2 or 3 drops of ammonia water. No precipitate should form within fifteen minutes.‡

POTASSIUM BICARBONATE

$KHCO_3$. Mol. Wt. 100.11

Colorless, transparent, rhombic prisms or plates, soluble in 4 parts of water.

TESTS OF PURITY

Sulphates. — On boiling the solution of 3 gm. of potassium bicarbonate in 50 c.c. of water and 6 c.c. of hydrochloric acid for several minutes, and then adding barium chloride solution, no precipitate of barium sulphate should form within fifteen hours. (Indicating less than 0.004 per cent SO_3.)

Chlorides. — Dissolve 3 gm. of potassium bicarbonate in 50 c.c. of water, add 10 c.c. of nitric acid and some silver nitrate solution. More than a slight opalescence should not develop. (Indicating less than 0.00075 per cent Cl.)

Nitrates. — Dissolve 3 gm. of potassium bicarbonate and a granule of sodium chloride in 10 c.c.of 16 per cent sulphuric acid; after adding to the solution 1 drop of a 1 : 1000 indigo solution and 10 c.c. of concentrated sulphuric acid, the blue color should not disappear on agitation. (Indicating less than 0.0011 per cent N_2O_5.)

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* The aqueous solution of potassium antimonate is used as a reagent for sodium. It is best to prepare the solution just before use by dissolving 1 gm. of the salt in 100 c.c. of boiling water, and filtering. The solutions to be tested for sodium should be neutral or slightly alkaline.

† A precipitate occurring in making these tests indicates that the preparation is unsuitable for use as a reagent for sodium.

‡ If a solution of 1 gm. of ammonium chloride in 10 c.c. of water is added to 20 c.c. of the 1 : 100 aqueous solution, a voluminous flocculent precipitate forms within a few minutes, if the addition of ammonia water is omitted.

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Silicates. — Dissolve 5 gm. of potassium bicarbonate in 20 c.c. of water and 15 c.c. of hydrochloric acid, and evaporate the solution on the water-bath in a platinum dish. Dry the residue for half an hour at 120° C., and then dissolve it in 25 c.c. of water with 3 c.c. of hydrochloric acid. The solution should be perfectly clear. (Indicating none present.)

Calcium, Aluminum, and Heavy Metals. — Dissolve 5 gm. of potassium bicarbonate in 25 c.c. of water and 15 c.c. of diluted acetic acid. Add 5 c.c. of ammonia water and heat for half an hour on the water-bath. No flocks should separate. (Indicating less than 0.04 per cent Al.) On adding to the solution some ammonium oxalate solution and ammonium sulphide solution, no change should appear. (Indicating less than 0.001 per cent Ca and no heavy metals present.)

Phosphates. — Dissolve 5 gm. of potassium bicarbonate in 50 c.c. of water, add to the solution 50 c.c. of nitric acid, 25 c.c. of ammonium molybdate solution, and heat at 30° to 40° C. for two hours. No yellow precipitate should form. (Indicating less than 0.004 per cent P_2O_5.)

Iron. — Dissolve 1 gm. of potassium bicarbonate in 3 c.c. of hydrochloric acid and 100 c.c. of water, add a drop of nitric acid and boil, then add potassium sulphocyanate solution. No red color should develop. (Indicating less than 0.0008 per cent Fe.)

Residue on Ignition. — 100 parts of potassium bicarbonate, on ignition, should leave 69 parts of residue. (Indicating 69 per cent.)

Quantitative Determination. —Titrate the solution of 1 gm. of potassium bicarbonate in 50 c.c. of water with normal hydrochloric acid solution, using methyl orange as indicator.

1 c.c. of normal HCl = 0.10011 gm. of $KHCO_3$, log. 00047.

POTASSIUM BINIODATE

KIO_3HIO_3. Mol. Wt. 389.95

Small, white crystals, clearly and completely soluble in 20 parts of cold water.

The preparation contains 100 per cent of KIO_3HIO_3.

TESTS OF PURITY

Chlorates. — Dissolve 0.2 gm. of potassium biniodate in 20 c.c. of water, add 20 c.c. of nitric acid and 10 c.c. of 1 : 20 solution of silver nitrate, and, after shaking thoroughly, filter. On adding to the filtrate 10 c.c. of about 35 per cent formaldehyde solution free from chlorides, and heating the mixture to boiling, neither a turbidity nor a precipitate should appear. (Indicating less than 0.15 per cent Cl_2O_5.)

Quantitative Determination. — The quantitative determination may be carried out either acidimetrically or iodometrically. The salt to be used for the quantitative determination must first be brought to constant weight at 98° C. in a drying-oven.

(*a*) For the acidimetric determination about 3.5 gm. of potassium biniodate are dissolved in 200 c.c. of water. The titration is made with fifth-normal solution of potassium hydroxide in the boiling solution, using phenolphthalein as indicator.

1 c.c. of normal KOH = 0.07799 gm. of KIO_3HIO_3, log. 89204.

(*b*) The iodometric determination is made as follows: Dissolve 0.10 to 0.15 gm. of potassium biniodate in 20 c.c. of water, add 3 gm. of potassium iodide

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and 5 c.c. of hydrochloric acid, then dilute the liquid with 100 c.c. of water, and titrate the liberated iodine with decinormal sodium thiosulphate solution.

1 c.c. of decinormal $Na_2S_2O_3 = 0.00324958$ gm. of KIO_3HIO_3, log. 51182.

Potassium biniodate may also be tested as to the correctness of its composition by comparing the iodine found in (*b*) with the quantity of iodine liberated from potassium biniodate by potassium iodide without the addition of an acid.

As will be seen from the equations given below, when potassium biniodate is decomposed by potassium iodide, without the addition of an acid, it liberates exactly one-twelfth of the quantity of iodine which is liberated if the same quantity of potassium biniodate is decomposed by potassium iodide in the presence of hydrochloric or sulphuric acid.

The determination of the quantity of iodine liberated, when the decomposition of the biniodate takes place in the absence of acid, is carried out as follows: To the solution of 1 gm. of potassium biniodate in 20 c.c. of water, add 3 gm. of perfectly neutral potassium iodide. Dilute with 100 c.c. of water, and titrate the liberated iodine with decinormal sodium thiosulphate solution. It is necessary in carrying out this determination to use water perfectly free from carbon dioxide, i.e., thoroughly boiled.

The iodine determined by this method, as well as that obtained under (*b*) and, of course, also the number of cubic centimeters required in the titrations, provided the same quantities of potassium biniodate are used, bear the ratio 1 : 12.

This determination is of special value because the correct composition of the preparation may be controlled even without knowing the exact titer of the sodium thiosulphate solution.

NOTE. — The course of decomposition of biniodate by potassium iodide in the absence of acid may be shown by the following equations:

1. $5KHI_2O_6 + 5KI = 10KIO_3 + 5HI$;
2. $KHI_2O_6 + 5HI = KIO_3 + 6I + 3H_2O$.

One KHI_2O_6, therefore, liberates one I.

In the presence of an acid, the reaction is as follows:

$KHI_2O_6 + 10KI + 11HCl = 11KCl + 6H_2O + 12I$.

Here, therefore, one KHI_2O_6 liberates twelve I.

POTASSIUM BISULPHATE

(Potassium Acid Sulphate)

$KHSO_4$. Mol. Wt. 136.18

Colorless crystals, easily soluble in water. The aqueous solution is acid to litmus paper.

TESTS OF PURITY

Heavy Metals. — The solution of 1 gm. of potassium bisulphate in 20 c.c. of water should not be affected by hydrogen sulphide water. On now adding ammonia water to alkalinity, and a few drops of ammonium sulphide solution, neither a precipitate nor a green or brown color should appear. (Indicating none present.)

Chlorides. — The 1 : 20 aqueous solution should not be rendered more than slightly opalescent on the addition of silver nitrate solution. (Indicating less than 0.002 per cent Cl.)

Arsenic. — The mixture of 1 gm. of finely powdered potassium bisulphate with 3 c.c. of stannous chloride solution should not acquire a dark color within one hour. (Indicating less than 0.0015 per cent As.)

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Quantitative Determination. — Dissolve 1 gm. of potassium bisulphate in 50 c.c. of water, and titrate with normal solution of potassium hydroxide, using methyl orange as the indicator.

1 c.c. of normal KOH = 0.13618 gm. of $KHSO_4$, log. 13411.

POTASSIUM BISULPHITE

(Potassium Acid Sulphite)

$KHSO_3$. Mol. Wt. 120.18

A white, crystalline powder, having an odor of sulphurous acid and easily soluble in water. The aqueous solution is acid to litmus paper.

TESTS OF PURITY

Chlorides. — Dissolve 1 gm. of potassium bisulphite in 10 c.c. of water, add 10 c.c. of perhydrol, make alkaline with a solution of potassium hydroxide purest, and evaporate on the water-bath. Dissolve the residue in 20 c.c. of water, acidulate with nitric acid, and add silver nitrate solution. At most a slight opalescence should appear. (Indicating less than 0.002 per cent Cl.)

Heavy Metals. — Dissolve 2 gm. of potassium bisulphite in 10 c.c. of water and 20 c.c. of hydrochloric acid, evaporate on the water-bath, dissolve the residue in 20 c.c. of water, and add to the solution hydrogen sulphide water. No change should appear, nor should a precipitate form on adding ammonia water and ammonium sulphide solution. (Indicating none present.)

Arsenic. — Introduce 5 gm. of potassium bisulphite, a little at a time, into 25 c.c. of nitric acid (sp.gr. 1.3), add 5 c.c. of concentrated sulphuric acid, and evaporate the mixture, first as far as possible on the water-bath and then upon the sand-bath, until sulphuric acid vapors begin to be evolved. Cool the residue and dissolve it in 50 c.c. of water. Set in operation a Marsh apparatus by means of 20 gm. of arsenic-free zinc and diluted (1 : 5) sulphuric acid, and add the solution in small portions to the generating flask of the apparatus. After one hour no deposit of arsenic should be visible in the reduction-tube. (Indicating less than 0.0002 per cent As.)

Quantitative Determination. — Dissolve 1 gm. of potassium bisulphite in boiled water and dilute to 100 c.c. Allow the solution to run from a burette into a mixture of 30 c.c. of decinormal iodine solution and 5 c.c. of hydrochloric acid, with constant shaking, until decolorization is complete.

1 c.c. of decinormal I = 0.00609 gm. of $KHSO_3$, log. 77880.

POTASSIUM BITARTRATE

(Potassium Acid Tartrate)

$KHC_4H_4O_6$. Mol. Wt. 188.14

A white, crystalline powder, soluble in about 200 parts of cold, and in 20 parts of boiling, water, and insoluble in alcohol. Potassium bitartrate is also soluble in a solution of sodium hydroxide, and in potassium carbonate solution with the evolution of carbon dioxide. The preparation contains 100 per cent of $KHC_4H_4O_6$.

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TESTS OF PURITY

Moisture. — 5 gm. of potassium bitartrate, when dried at 100° C., should not suffer any loss in weight. (Indicating none present.)

Chlorides. — Dissolve 1 gm. of potassium bitartrate in 20 c.c. of water and add 5 c.c. of nitric acid. The solution must not be rendered more than slightly opalescent on the addition of silver nitrate solution. (Indicating less than 0.002 per cent Cl.)

Sulphates. — Dissolve 1 gm. of potassium bitartrate in 20 c.c. of water, add 5 c.c. of nitric acid and barium nitrate solution. No precipitate should form within fifteen hours. (Indicating less than 0.035 per cent SO_3.)

Ammonium Compounds. — On heating 2 gm. of potassium bitartrate with 10 c.c. of sodium hydroxide solution no vapors of ammonia should be evolved (to be ascertained by moist litmus paper). (Indicating less than 0.00175 per cent NH_3.)

Calcium. — Dissolve 1 gm. of potassium bitartrate in 5 c.c. of diluted acetic acid and 25 c.c. of water, with the aid of heat. Allow to become perfectly cold, filter, and to the filtrate add a few drops of ammonium oxalate solution. The liquid should show no turbidity within ten minutes. (Indicating less than 0.025 per cent Ca.)

Heavy Metals.* — The solution of 5 gm. of potassium bitartrate in 25 c.c. of water and 25 c.c. of ammonia water should show no change on the addition of hydrogen sulphide water. (Indicating none present.)

Quantitative Determination. — (*a*) Dissolve 5 gm. of potassium bitartrate in 250 c.c. of hot water, and while boiling titrate the solution with normal solution of potassium hydroxide, using phenolphthalein as the indicator.

1 c.c. of normal KOH = 0.18814 gm. of $KHC_4H_4O_6$, log. 27448.

(*b*) Cautiously incinerate 3 gm. of potassium bitartrate in a platinum crucible. When cold, dissolve the contents of the crucible in water, and titrate with normal hydrochloric acid solution, using methyl orange as the indicator.

1 c.c. of normal HCl = 0.18814 gm. of $KHC_4H_4O_6$, log. 27448.

POTASSIUM BROMATE

$KBrO_3$. Mol. Wt. 167.02

White crystals or crystalline powder, soluble in 15 parts of cold, or 2 parts of boiling, water. The aqueous solution is neutral to litmus paper. The preparation contains 100 per cent of $KBrO_3$.

TESTS OF PURITY

Potassium Bromide. — Dissolve 2 gm. of potassium bromate in 30 c.c. of water and add diluted sulphuric acid. The solution should not immediately acquire a yellow color. (Indicating less than 0.04 per cent KBr.)

Quantitative Determination. — For this purpose, the salt must be dried over sulphuric acid. Dissolve 0.10 to 0.15 gm. of the dried potassium bromate in 20 c.c. of water, add 3 gm. of potassium iodide and 5 c.c. of hydrochloric acid, and titrate the liberated iodine with decinormal sodium thiosulphate solution.

1 c.c of decinormal $Na_2S_2O_3$ = 0.00278366 gm. of $KBrO_3$, log. 44461.

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* Regarding the examination of potassium bitartrate for lead, see Tatlock and Thompson, Analyst, **33**, 173 (1908); *abst.*, C. A., **2**, 2269 (1908).

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POTASSIUM BROMIDE

KBr. Mol. Wt. 119.02

White, cubical, lustrous crystals, permanent in the air; soluble in 2 parts of water and in about 200 parts of 85 per cent alcohol.

TESTS OF PURITY

Potassium Carbonate. — Powdered potassium bromide should not immediately render red litmus paper blue, and the 1 : 20 aqueous solution should not be reddened on the addition of phenolphthalein solution. (Indicating less than 0.04 per cent K_2CO_3.)

Potassium Bromate. — The 1 : 10 aqueous solution of potassium bromide should not become colored on the addition of diluted sulphuric acid; chloroform shaken with this mixture should not become yellow. (Indicating less than 0.004 per cent $KBrO_3$.)

Heavy Metals, Sulphates, and Barium Salts. — 20 c.c. portions of the 1 : 20 solution should show no change with hydrogen sulphide water (indicating no heavy metals present), or with barium nitrate solution (indicating less than 0.00125 per cent SO_3), or with diluted sulphuric acid. (Indicating less than 0.002 per cent Ba.)

Iodides. — Add 3 drops of ferric chloride solution and some starch solution to the 1 : 20 aqueous solution of potassium bromide. No blue coloration should take place within ten minutes. (Indicating less than 0.1 per cent I.)

Quantitative Determination and Test for Excess of Potassium Chloride.* — On adding a few drops of potassium chromate solution to 50 c.c. of the aqueous solution, prepared from 3 gm. of potassium bromide, dried at 100° C. before weighing, dissolved, and diluted with water to 500 c.c., the solution should require not more than 25.4 c.c. and not less than 25.1 c.c. of decinormal silver nitrate solution to produce a permanent red color. (Indicating not more than 1.3 per cent KCl.)

1 c.c. of decinormal $AgNO_3$ = 0.011902 gm. KBr, log. 07562.

POTASSIUM CARBONATE

K_2CO_3. Mol. Wt. 138.3

A white, granular, hygroscopic powder of alkaline reaction, soluble in 1 part of water, but insoluble in absolute alcohol. The preparation should contain at least 99 per cent of K_2CO_3.

TESTS OF PURITY

Heavy Metals. — 20 c.c. of the 1 : 20 aqueous solution should show no change of color on the addition of hydrogen sulphide water. Nor should any change of color be shown upon acidulating 20 c.c. of the 1 : 20 solution with acetic acid and then adding hydrogen sulphide water. (Indicating none present.)

Chlorides. — The solution of 1 gm. of potassium carbonate in 20 c.c. of water,

* Potassium chloride is almost always present in potassium bromide, though usually in very small quantities only.

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acidified with nitric acid, should afford at most a slight opalescence on the addition of silver nitrate solution. (Indicating less than 0.002 per cent Cl.)

Sulphates. — On acidifying 20 c.c. of the 1 : 20 aqueous solution with hydrochloric acid, then boiling for a few minutes and adding barium chloride solution, no precipitate of barium sulphate should form within fifteen hours. (Indicating less than 0.0075 per cent SO_3.)

Nitrates. — Dissolve 0.2 gm. of potassium carbonate in 2 c.c. of diluted sulphuric acid, and mix the fluid with 2 c.c. of concentrated sulphuric acid. On now cooling the solution, and overlaying it with 1 c.c. of a ferrous sulphate solution, no colored zone should form at the contact-surfaces of the two liquids. (Indicating less than 0.08 per cent N_2O_5.)

Potassium Cyanide. — Dissolve 0.5 gm. of ferrous sulphate in 5 c.c. of water, add 5 c.c. of a 1 : 20 potassium carbonate solution, and 1 or 2 drops of ferric chloride solution. Heat to about 60 to 70° C., and acidulate with hydrochloric acid. No green color should develop, nor should a blue precipitate form. (Indicating less than 0.048 per cent CN.)

Sulphides, Sulphites, and Thiosulphates. — On pouring 1 c.c. of the 1 : 20 aqueous solution into 10 c.c. of decinormal silver nitrate solution, a yellowish-white precipitate forms, which should not become darker (gray to brownish to black) on being heated to 60° to 70° C. (Indicating less than 0.02 per cent S, 0.008 per cent SO_2, or 0.08 per cent $K_2S_2O_3$.)

Phosphates. — Dissolve 5 gm. of potassium carbonate in 50 c.c. of water, acidulate the solution with 50 c.c. of nitric acid, and add 25 c.c. of ammonium molybdate solution. No yellow precipitate should form on standing two hours at about 40° C. (Indicating less than 0.004 per cent P_2O_5.)

Silicates. — Dissolve 5 gm. of potassium carbonate in a platinum dish in 20 c.c. of hydrochloric acid and 20 c.c. of water, and evaporate to dryness. Dry the residue for half an hour at about 120° C., and then dissolve it in 3 c.c. of hydrochloric acid and 25 c.c. of water. The solution must be perfectly clear. (Indicating none present.)

Aluminum and Calcium. — Dissolve 5 gm. of the carbonate in 25 c.c. of water and 25 c.c. of acetic acid (sp.gr. 1.041), add 12 c.c. of ammonia water, and heat for half an hour on the water-bath. No flocculent precipitate should form, (indicating less than 0.04 per cent Al), nor should any change take place on the addition of ammonium oxalate solution. (Indicating less than 0.001 per cent Ca.)

Quantitative Determination. — Dissolve 1 gm. of potassium carbonate in 50 c.c. of water, and titrate with normal solution of hydrochloric acid, using methyl orange as indicator. At least 14.3 c.c. of the hydrochloric acid solution should be required to produce the red end-point.

1 c.c. of normal HCl = 0.0691 gm. of K_2CO_3, log. 83948.

POTASSIUM CARBONATE SOLUTION

A clear, colorless, alkaline liquid, of specific gravity 1.334 to 1.338. 100 parts contain about 33 parts of potassium carbonate.

TESTS OF PURITY

The tests to be made are those given under Potassium Carbonate. But use 3 c.c. of potassium carbonate solution for each gram of potassium carbonate.

Quantitative Determination. — Dilute 5 gm. of potassium carbonate solution with 30 c.c. of water and titrate with normal solution of hydrochloric acid, using methyl orange as indicator. At least 23.7 c.c. of the acid solution should be required to effect the color change.

1 c.c. of normal HCl = 0.0691 gm. of K_2CO_3, log. 83948.

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POTASSIUM CHLORATE

$KClO_3$. Mol. Wt. 122.56

Colorless, lustrous, tabular, crystals soluble in 17 parts of cold, and in 2 parts of boiling, water, and in 130 parts of about 85 per cent (by weight) alcohol. The salt is practically insoluble in absolute alcohol and in ether. The aqueous solution is neutral.

TESTS OF PURITY

Chlorides. — 20 c.c. of the 1 : 20 aqueous solution should not be affected by silver nitrate solution. (Indicating less than 0.001 per cent Cl.)

Heavy Metals. — The solution of 3 gm. of potassium chlorate in 30 c.c. of warm water should be perfectly clear, and should remain unchanged on the addition of hydrogen sulphide water. (Indicating none present.)

Calcium. — The solution of 1 gm. of potassium chlorate in 20 c.c. of water should show no change on adding ammonium oxalate solution. (Indicating less than 0.005 per cent Ca.)

Bromates. — Gently ignite 1 gm. of potassium chlorate, dissolve the residue in water, and add to the solution a few drops of chlorine water. On shaking this mixture with chloroform, the latter should not become colored yellow or brown. (Indicating less than 0.08 per cent Br_2O_5.)

Nitrates. — On heating 1 gm. of potassium chlorate with 5 c.c. of sodium hydroxide solution (sp.gr. 1.3), and a mixture of 0.5 gm. of zinc dust and 0.5 gm. of powdered iron, no ammonia should be evolved (to be ascertained by moist litmus paper). (Indicating less than 0.0016 per cent N_2O_5.)

Sulphates. — On adding barium chloride solution to 20 c.c. of the 1 : 20 aqueous solution, no precipitate of barium sulphate should form on standing fifteen hours. (Indicating less than 0.001 per cent SO_3.)

Arsenic. — Pour 150 c.c. of hydrochloric acid over 20 gm. of potassium chlorate in a porcelain dish. As soon as the evolution of chlorine has slackened, heat the solution on the water-bath until free from the odor of chlorine. A Marsh apparatus is set in operation, containing 20 gm. of arsenic-free, granulated zinc and dilute (1 : 5) sulphuric acid. The solution obtained above, cooled and diluted with water, is then introduced into the generating flask of the apparatus in small portions at a time. No deposit of arsenic should be visible in the reduction tube within one hour. (Indicating less than 0.00005 per cent As.)

Quantitative Determination.* — Into a glass-stoppered flask of 400 to 500 c.c. capacity introduce 50 c.c. of hydrochloric acid, replace the air in the flask by carbon dioxide, add a solution of 5 gm. of potassium iodide in 10 c.c. of boiled and cooled water, and finally add a solution of 1 gm. of the potassium chlorate in 100 c.c. of water. Close the flask, let the mixture stand one hour in a dark place, dilute it with 200 c.c. of boiled and cooled water and then titrate with decinormal sodium thiosulphate solution.

1 c.c. of decinormal $Na_2S_2O_3 = 0.00204266$ gm. of $KClO_3$, log, 31019.

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* A. Kolb and E. Davidson, Z. angew. Chem., **17**, 1883 (1904); *abst.*, J. Chem. Soc., 88, II, 59 (1904).

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POTASSIUM CHLORIDE

KCl. Mol. Wt. 74.56

Colorless, cubical crystals, or white, crystalline powder, soluble in 3 parts of cold water; insoluble in absolute alcohol or ether. The aqueous solution is neutral.

TESTS OF PURITY

Heavy Metals and Alkaline Earths. — The solution of 3 gm. of potassium chloride in 50 c.c. of water should not be affected by ammonium oxalate solution; or by sodium carbonate solution; or by ammonium sulphide solution. (Indicating no heavy metals present, and less than 0.01 per cent earths as Ca.)

Sulphates. — On adding barium chloride solution to 20 c.c. of the 1 : 20 aqueous solution, no precipitate of barium sulphate should form on standing fifteen hours. (Indicating less than 0.001 per cent SO_3.)

Nitrates. — Dissolve 1 gm. of potassium chloride in 10 c.c. of water and add 1 drop of 1 : 1000 indigo solution and 10 c.c. of concentrated sulphuric acid; on shaking, the blue color of the mixture should not disappear. (Indicating less than 0.0032 per cent N_2O_5.)

Chlorates. — 20 c.c. of the 1 : 20 solution of potassium chloride should not be colored blue on the addition of zinc iodide-starch solution and hydrochloric acid. (Indicating less than 0.018 per cent Cl_2O_5.)

Magnesium. — Dissolve 1 gm. of potassium chloride in 5 c.c. of water and add 5 c.c. of ammonia water and some ammonium phosphate solution. No precipitate should form on standing fifteen hours. (Indicating less than 0.002 per cent Mg.)

POTASSIUM CHROMATE

(Yellow Potassium Chromate)

K_2CrO_4. Mol. Wt. 194.2

Yellow, rhombic crystals, permanent in the air, soluble in 2 parts of cold water. The 1 : 20 aqueous solution is slightly alkaline to litmus paper.

TESTS OF PURITY

Free Alkali. — The solution of 0.1 gm. of potassium chromate in 25 c.c. of water should not acquire a red color on the addition of a few drops of phenolphthalein solution. (Indicating less than 0.2 per cent as KOH.)

Sulphates. — Dissolve 3 gm. of potassium chromate in 100 c.c. of water and add 30 c.c. of hydrochloric acid, followed by barium chloride solution. No precipitate of barium sulphate should form within fifteen hours. (Indicating less than 0.029 per cent SO_3.)

Chlorides. — Dissolve 1 gm. of potassium chromate in 20 c.c. of water, add to the solution 10 c.c. of nitric acid, and heat the mixture to about 50° C. On now adding a few drops of silver nitrate solution, no turbidity or precipitate should form within five minutes. (Indicating less than 0.0025 per cent Cl.)

Alumina and Alkaline Earths. — Dissolve 2 gm. of potassium chromate in 30 c.c. of water, add 5 c.c. of ammonia water and some ammonium oxalate

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solution. No precipitate should form within fifteen hours. (Indicating less than 0.1 per cent Al and less than 0.005 per cent earths as Ca.)

Quantitative Determination. — Dissolve 1 gm. of potassium chromate in water and dilute to 100 c.c. Introduce 10 c.c. of this solution into a glass-stoppered flask of about 400 c.c. capacity, add 2 gm. of potassium iodide, 5 c.c. of 16 per cent sulphuric acid, and 350 c.c. of thoroughly boiled water. Titrate the liberated iodine with decinormal sodium thiosulphate solution, using starch solution as the indicator.

1 c.c. of decinormal $Na_2S_2O_3 = 0.0064733$ gm. of K_2CrO_4, log. 81113.

POTASSIUM CYANIDE

KCN. Mol. Wt. 65.11

A white powder, easily soluble in 2 parts of water, difficultly soluble in absolute alcohol, but readily in boiling 60 per cent alcohol. The aqueous solution is alkaline to litmus paper. The salt contains at least 96 per cent of KCN.

TESTS OF PURITY

Sulphides. — On adding a solution of lead acetate to the solution of 1 gm. of potassium cyanide in 20 c.c. of water, the resulting precipitate should have a pure white color. (Indicating less than 0.003 per cent S.)

Carbonates, Sulphocyanates, and Ferrocyanides. — On adding 5 c.c. of hydrochloric acid to the solution of 1 gm. of potassium cyanide in 20 c.c. of water—which should be done under a hood with a good draft—only a slight effervescence should be perceptible. (Indicating less than 4 per cent CO_2.) On adding a drop of ferric chloride solution to the acid liquid, neither a red nor a blue color should develop. (Indicating less than 0.015 per cent CNS, or 0.005 per cent $Fe[CN]_6$.)

Sulphates. — On adding to 20 c.c. of the 1 : 20 aqueous solution 5 c.c. of hydrochloric acid, and some barium chloride solution, no turbidity should develop. (Indicating less than 0.0875 per cent SO_3.)

Chlorides. — Dissolve 0.5 gm. of potassium cyanide in 20 c.c. of water and add 30 c.c. of 1 : 20 silver nitrate solution and 10 c.c. of nitric acid. Filter off the precipitate, wash it with water, dry it at 100° C., remove it from the filter and ignite it in a porcelain crucible. Add to the residue a few pieces of zinc and 20 c.c. of 16 per cent sulphuric acid, let the mixture stand two to three hours, shaking frequently, and filter. On adding to the filtrate nitric acid and silver nitrate solution, not more than a slight opalescence should develop. (Indicating less than 0.004 per cent Cl.)

Quantitative Determination. — Dissolve 1 gm. of potassium cyanide in water and dilute to 100 c.c. Dilute 10 c.c. of this solution with 90 c.c. of water, add a granule of sodium chloride, and titrate with decinormal silver nitrate solution until a permanent, whitish turbidity appears.

1 c.c. of decinormal $AgNO_3 = 0.013022$ gm. of KCN, log. 11467.

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POTASSIUM DICHROMATE

(Potassium Bichromate)

$K_2Cr_2O_7$. Mol. Wt. 294.2

Dark, yellowish-red, triclinic prisms or plates, soluble in 10 parts of cold, or in about 1.5 parts of boiling, water. The aqueous solution reddens blue litmus paper.

TESTS OF PURITY

Sulphates. — Dissolve 3 gm. of potassium dichromate in 100 c.c. of water, and add 30 c.c. of hydrochloric acid followed by barium chloride solution. No precipitate of barium sulphate should form on standing fifteen hours. (Indicating less than 0.029 per cent SO_3.)

Chlorides. — Dissolve 1 gm. of potassium dichromate in 20 c.c. of water, add 10 c.c. of nitric acid, and heat the mixture to about 50° C. On now adding a few drops of silver nitrate solution, no turbidity should develop within five minutes. (Indicating less than 0.0025 per cent Cl.)

Alumina and Alkaline Earths. — Dissolve 2 gm. of potassium dichromate in 30 c.c. of water, make alkaline with 10 c.c. of ammonia water, and add ammonium oxalate solution. No precipitate should form within fifteen hours. (Indicating less than 0.1 per cent Al, and less than 0.005 per cent earths as Ca.)

Quantitative Determination. — Dissolve 1 gm. of potassium dichromate in water, dilute to 100 c.c., and introduce 10 c.c. of this solution into a glass-stoppered flask of about 400 c.c. capacity, together with 2 gm. of potassium iodide and 5 c.c. of 16 per cent sulphuric acid. Dilute with 350 c.c. of thoroughly boiled water, and titrate the liberated iodine with decinormal sodium thiosulphate solution, using starch solution as the indicator.

1 c.c. of decinormal $Na_2S_2O_3 = 0.0049033$ gm. of $K_2Cr_2O_7$, log. 69049.

POTASSIUM FERRICYANIDE

(Red Prussiate of Potash)

$K_3Fe(CN)_6$. Mol. Wt. 329.2

Ruby-red, shining crystals, soluble in 2.5 parts of cold, and in 1.5 parts of boiling, water. The 1 : 20 aqueous solution is neutral.

TESTS OF PURITY

Ferrous Salts. — Dissolve 2 gm. of potassium ferricyanide in 400 c.c. of water and 5 c.c. of 16 per cent sulphuric acid, and titrate the solution with decinormal solution of potassium permanganate; at most 0.2 c.c. of the permanganate solution should be necessary to effect a color change from yellowish-green to yellowish-red. In order better to observe the color change, compare the color of the solution that has been titrated with that of another solution of 2 gm. of the ferricyanide in 400 c.c. of water and 5 c.c. of 16 per cent sulphuric acid. (Indicating not more than 0.056 per cent Fe″.)

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Sulphates. — On adding 1 c.c. of hydrochloric acid to 20 c.c. of the 1 : 20 aqueous solution, and then adding barium chloride solution, no precipitate of barium sulphate should form on standing fifteen hours. (Indicating less than 0.005 per cent SO_3.)

Chlorides. — Deflagrate a mixture of 0.5 gm. of powdered potassium ferricyanide and 1 gm. of potassium nitrate* by introducing small quantities at a time into a porcelain crucible heated to redness. Ignite for a few minutes and leach the melt with 20 c.c. of water. Filter, to the filtrate add 0.5 gm. of potassium nitrate, evaporate to dryness, re-fuse in a porcelain crucible until quiescent, and allow to cool. When cold, dissolve in 20 c.c. of water, add 3 c.c. of nitric acid and silver nitrate solution. The liquid should show at most an opalescence. (Indicating less than 0.01 per cent Cl.)

Quantitative Determination. — Dissolve 0.7 gm. of potassium ferricyanide in 50 c.c. of water, add 3 gm. of potassium iodide and 1.5 gm. of zinc sulphate, free from iron, shake thoroughly, and titrate the free iodine with decinormal sodium thiosulphate solution, using starch solution as indicator.

1 c.c. of decinormal $Na_2S_2O_3 = 0.03292$ gm. of $K_3Fe(CN)_6$, log. 51746.

POTASSIUM FERROCYANIDE

(Yellow Prussiate of Potash)

$K_4Fe(CN)_6+3H_2O$. Mol. Wt. 422.35

Citron-yellow, tabular crystals, which are quite permanent in the air and are soluble in 4 parts of cold, and 2 parts of boiling, water; insoluble in alcohol. The 1 : 20 aqueous solution is neutral.

TESTS OF PURITY

Carbonates. — On treating 1 gm. of powdered potassium ferrocyanide with dilute sulphuric acid, there should be no evolution of gas. (Indicating less than 0.75 per cent CO_2.)

Sulphates. — Dissolve 1 gm. of potassium ferrocyanide in 20 c.c. of water, and add 1 c.c. of hydrochloric acid followed by barium chloride solution. The solution should not be affected. (Indicating less than 0.0075 per cent SO_3.)

Chlorides. — Deflagrate a mixture of 0.5 gm. of powdered potassium ferrocyanide and 1 gm. of potassium nitrate,* by introducing small quantities at a time into a porcelain crucible heated to redness. Treat the residue with 20 c.c. of water, filter, to the filtrate add 0.5 gm. of potassium nitrate, and evaporate to dryness in a porcelain crucible. Fuse the residue until quiescent, allow to cool, dissolve the melt in 20 c.c. of water, and add 3 c.c. of nitric acid and silver nitrate solution. The liquid should not develop more than an opalescence. (Indicating less than 0.01 per cent Cl.)

Quantitative Determination. — Dissolve 1 gm. of potassium ferrocyanide in 200 c.c. of water, add 10 c.c. of concentrated sulphuric acid, and titrate with decinormal potassium permanganate solution until the color changes from yellowish-green to yellowish-red.

1 c.c. of decinormal $KMnO_4 = 0.042235$ gm. of $K_4Fe(CN)_6+3H_2O$, log. 62567.

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* The potassium nitrate should be tested for chloride and perchlorate. See under Potassium Nitrate.

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POTASSIUM HYDROXIDE

(Caustic Potash; Potassium Hydrate)

KOH. Mol. Wt. 56.11

In the laboratory, three grades of potassium hydroxide are generally used:

1. Potassium Hydroxide, Purest.
2. Potassium Hydroxide, Pure.
3. Potassium Hydroxide, Purified.

These three preparations differ chiefly in their content of chloride, sulphate, silicate, and alumina.

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POTASSIUM HYDROXIDE, PUREST

White, very hygroscopic pieces showing a crystalline structure on fracture. The preparation contains 82 to 85 per cent of KOH.

TESTS OF PURITY

Sulphates. — Dissolve 3 gm. of potassium hydroxide in 50 c.c. of water, add 10 c.c. of hydrochloric acid, heat to boiling, and then add barium chloride solution. No precipitate of barium sulphate should form on standing fifteen hours. (Indicating less than 0.004 per cent SO_3.)

Chlorides. — Dissolve 1 gm. of potassium hydroxide in 20 c.c. of water, and add 5 c.c. of nitric acid, followed by a few drops of silver nitrate solution. Not more than a slight opalescent turbidity should develop within one minute (Indicating less than 0.0025 per cent Cl.)

Nitrates. — Dissolve 2 gm. of potassium hydroxide and a granule of sodium chloride in 10 c.c. of 16 per cent sulphuric acid, and add 1 drop of 1 : 1000 indigo solution and 10 c.c. of concentrated sulphuric acid. The blue color of the mixture should not disappear on agitation. (Indicating less than 0.0016 per cent N_2O_5.)

Nitrogen as Nitrates, Nitrites, Ammonia, etc. — Dissolve 50 gm. of potassium hydroxide in 100 c.c. of water in a distillation flask. To the solution add 2 gm. of Devarda's metal, connect the flask with a bulb tube, condenser, and a receiver containing 2 to 3 c.c. of fifth-normal hydrochloric acid solution and 10 c.c. of water; allow to stand one to two hours, and then distil off about 50 c.c. Titrate the distillate with fifth-normal potassium hydroxide solution, using methyl orange as the indicator. At most 0.2 c.c. of fifth-normal hydrochloric acid solution should have been required to neutralize the ammonia. (Indicating not more than 0.00112 per cent N.)

Nitrites. — The solution of 1 gm. of potassium hydroxide in 10 c.c. of water and 10 c.c. of diluted sulphuric acid should not be colored blue on adding zinc iodide-starch solution. (Indicating less than 0.00011 per cent N_2O_3.)

Phosphates. — Dissolve 5 gm. of potassium hydroxide in 50 c.c. of water and add 30 c.c. of nitric acid, followed by 25 c.c. of a solution of ammonium

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molybdate in nitric acid. This mixture should deposit no yellow precipitate on standing two hours at about 40° C. (Indicating less than 0.004 per cent P_2O_5.)

Silicates. — Dissolve 5 gm. of potassium hydroxide in 25 c.c. of water in a platinum dish, add 25 c.c. of hydrochloric acid, and then evaporate to dryness on the water-bath. Dry the residue for half an hour at about 120° C., and then dissolve it in 10 c.c. of hydrochloric acid and 90 c.c. of water. Any insoluble residue should be filtered off, washed, and ignited. Its weight should not exceed 0.001 gm. (Indicating not more than 0.02 per cent SiO_2.)

Aluminum, Calcium, and Heavy Metals.— 5 gm. of potassium hydroxide should yield a clear and colorless solution with 10 c.c. of water. To this solution add 25 c.c. of acetic acid (sp.gr. 1.041), followed by 10 c.c. of ammonia water, dilute with 55 c.c. of water, and heat the fluid on the water-bath until the odor of ammonia has disappeared. Now add a few drops of ammonia water, and allow to stand fifteen hours. At the end of this time there should be no precipitate, or, at most, only an exceedingly slight, flocculent precipitate. In the latter case the precipitate is collected by filtration, washed, and ignited. Its weight should not exceed 0.001 gm. (Indicating not over 0.0106 per cent Al.) The following tests are now carried out with the filtrate:

To 50 c.c. add ammonium oxalate solution. No precipitate of calcium oxalate should form within two hours. (Indicating less than 0.012 per cent Ca.)

To 50 c.c. add a few drops of ammonium sulphide solution. Neither a green or a brown color nor a precipitate should appear. (Indicating no heavy metals present.)

Substances Insoluble in Alcohol. — Dissolve 5 gm. of potassium hydroxide in 10 c.c. of water and mix the solution with 50 c.c. of 85 per cent alcohol. There should be no precipitate within one hour. (Indicating none present.)

Quantitative Determination, and Determination of the Potassium Carbonate Content. — Titrate a cold solution of 1 gm. of potassium hydroxide in 100 c.c. of water with normal solution of hydrochloric acid, using phenolphthlein as the indicator. At least 14.8 c.c. of the acid should be necessary to destroy the red color. (First titration.) Now add 1 drop of methyl orange, and titrate further until the color again changes to red. In this second titration, not more than 0.2 c.c. of the acid should be required. (Indicating not more than 2.76 per cent K_2CO_3).*

1 c.c. of normal HCl = 0.05611 gm. of KOH, log. 74904.

1 c.c. of normal HCl = 0.0691 gm. of K_2CO_3, log. 83948.

II

POTASSIUM HYDROXIDE, PURE

White, very hygroscopic sticks or pieces, exhibiting a crystalline fracture. The preparation contains at least 80 per cent of KOH.

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* From the results of both titrations, the content of potassium hydroxide and potassium carbonate are calculated as follows:

The result of the second titration multiplied by two gives the number of cubic centimeters which are necessary to neutralize the potassium carbonate originally present.

On subtracting the number of cubic centimeters required in the second titration from the number of cubic centimeters used in the first titration, the remainder gives the number of cubic centimeters which were required to neutralize the potassium hydroxide.

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TESTS OF PURITY

Sulphates, Nitrates and Nitrogen as Nitrates, Nitrites, Ammonia, etc. — The tests are carried out as detailed under Potassium Hydroxide, Purest.

Chlorides.—Dissolve 1 gm. of potassium hydroxide in 20 c.c. of water, and add 5 c.c. of nitric acid and a few drops of silver nitrate solution. An opalescence may develop, but no precipitate should form immediately. (Indicating less than 0.005 per cent Cl.)

Silicates. — Dissolve 5 gm. of potassium hydroxide in 25 c.c. of water and 25 c.c. of hydrochloric acid in a platinum dish, and evaporate to dryness on the water-bath. Dry the residue for half an hour at about 120° C., then treat it with 10 c.c. of hydrochloric acid and 90 c.c. of water, filter, wash, and ignite any undissolved residue. The weight of the latter should not exceed 0.003 gm. (Indicating not more than 0.06 per cent SiO_2.)

Aluminum, Calcium, and Heavy Metals. — 5 gm. of potassium hydroxide should completely dissolve in 20 c.c. of water, affording a clear and colorless solution. Add to the solution 25 c.c. of acetic acid (sp.gr. 1.041) followed by 10 c.c. of ammonia water, dilute with 55 c.c. of water, heat the solution on the water-bath until the odor of ammonia has disappeared, and add a few drops more of ammonia water. On standing fifteen hours only slight precipitation should occur. The precipitate is filtered off, washed and ignited. Its weight should not exceed 0.003 gm. (Indicating not more than 0.032 per cent Al.) No precipitate should appear within two minutes on the addition of ammonium oxalate solution to the filtrate (indicating less than 0.04 per cent Ca); and the addition of ammonium sulphide solution should cause at most a slight green coloration, but in no case should a brown color or a precipitate appear. (Indicating at most a trace of heavy metals.)

Quantitative Determination, and Determination of the Potassium Carbonate Content. — Dissolve 1 gm. of potassium hydroxide in 100 c.c. of water, and titrate the cold solution with normal solution of hydrochloric acid, using phenolphthalein as the indicator. At least 14.5 c.c. of the acid should be necessary to destroy the red color. Now add 1 drop of methyl orange solution, and titrate further until the color again changes to red. In this second titration not more than 0.30 c.c. of the acid should be required. (Indicating not more than 4.15 per cent K_2CO_3).*

1 c.c. of normal HCl = 0.05611 gm. of KOH, log. 74904.

1 c.c. of normal HCl = 0.0691 gm. of K_2CO_3, log. 83948.

III

POTASSIUM HYDROXIDE, PURIFIED

White, very hygroscopic sticks or pieces which exhibit a crystalline fracture. The preparation should contain at least 80 per cent of KOH.

TESTS OF PURITY

Nitrates. — Dissolve 2 gm. of potassium hydroxide and a granule of sodium chloride in 10 c.c. of diluted sulphuric acid, and add 1 drop of 1 : 1000 indigo solution and 10 c.c. of concentrated sulphuric acid. The blue color of the mixture should not disappear on agitation. (Indicating less than 0.0016 per cent N_2O_5.)

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* See the note under Potassium Hydroxide, Purest.

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Aluminum, Calcium, and Heavy Metals. — 2.5 gm. of potassium hydroxide should completely dissolve in 10 c.c. of water, yielding a clear and colorless solution. Dilute the solution with 50 c.c. of water and add 15 c.c. of acetic acid (sp.gr. 1.041), followed by 10 c.c. of ammonia water; no precipitate should form. (Indicating less than 0.1 per cent Al.) The solution so tested should not exhibit an immediate turbidity on adding ammonium oxalate solution (indicating less than 0.1 per cent Ca); and on the addition of ammonium sulphide solution it should acquire at most a slight green color. (Indicating at most a trace of heavy metals.)

Quantitative Determination and Determination of the Potassium Carbonate Content. — The determinations are made as detailed under Potassium Hydroxide, Pure. The content of potassium carbonate should not exceed 5 per cent.

POTASSIUM HYDROXIDE SOLUTIONS

A clear, colorless liquid, of specific gravity 1.3, and containing about 32 per cent of potassium hydroxide.

Also a similar solution having a specific gravity 1.138 to 1.140 and containing about 15 per cent of potassium hydroxide.

TESTS OF PURITY

The tests and the quantitative determination, as given under Potassium Hydroxide, Pure, are to be made on each solution. But, of the solution having sp.gr. 1.3, use 2 c.c. (2.6 gm.) in place of each gram of the solid hydroxide referred to. And, of the solution having sp.gr. 1.138 to 1.140, use 5 c.c. (5.7 gm.).

POTASSIUM IODATE

KIO_3. Mol. Wt. 214.02

A white, crystalline powder, soluble in 13 parts of cold, and in 5 parts of boiling, water. The 1 : 20 aqueous solution should be clear and neutral to litmus paper. The preparation contains 100 per cent of KIO_3.

TESTS OF PURITY

Free Acids. — Dissolve about 0.5 gm. of potassium iodate in about 20 c.c. of boiled water, and add a small crystal of neutral potassium iodide and a few drops of starch solution. The liquid should not acquire a blue color within thirty seconds. (Indicating less than 0.07 per cent as HIO_3.)

Iodides. — Dissolve 1 gm. of potassium iodate in 20 c.c. of water, add 3 to 5 drops of diluted sulphuric acid, and shake the mixture with chloroform. The latter should not acquire a violet color. (Indicating less than 0.0015 per cent I.)

Chlorates. — This test is to be carried out as described under Potassium Biniodate. (Indicating less than 0.15 per cent Cl_2O_5.)

Quantitative Determination. — Dissolve 0.10 to 0.15 gm. of potassium iodate previously dried over sulphuric acid, in 20 c.c. of water, add 3 gm. of potassium iodide and 5 c.c. of hydrochloric acid, and titrate the liberated iodine with decinormal sodium thiosulphate solution.

1 c.c. of decinormal $Na_2S_2O_3 = 0.003567$ gm. of KIO_3, log. 55230.

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POTASSIUM IODIDE

KI. Mol. Wt. 166.02

White, cubical crystals, not becoming moist on contact with air. Potassium iodide is soluble in about 0.75 part of water, in about 12 parts of 85 per cent alcohol, and in 40 parts of absolute alcohol.

TESTS OF PURITY

Potassium Carbonate. — Potassium iodide when crushed and placed upon moistened red litmus paper should not immediately color the latter violet-blue.

On adding to a solution of 0.5 gm. of potassium iodide in 10 c.c. of water 1 drop of phenolphthalein solution, no red color should develop. (Indicating less than 0.08 per cent K_2CO_3.)

Heavy Metals. — 20 c.c. of the 1 : 20 aqueous solution of potassium iodide should afford no reaction with hydrogen sulphide water; and on further addition of ammonia water neither a green or brown color should develop, nor should a precipitate form. (Indicating none present.)

Sulphates. — 20 c.c. of the 1 : 20 aqueous solution should afford no reaction with barium chloride solution. (Indicating less than 0.01 per cent SO_3.)

Cyanides. — On adding to 20 c.c. of the 1 : 20 aqueous solution a granule of ferrous sulphate, 1 drop of ferric chloride solution, and 5 c.c. of sodium hydroxide solution (sp.gr. 1.3), then heating the mixture to 50° to 60° C., and adding 10 c.c. of hydrochloric acid, no blue color should develop. (Indicating less than 0.03 per cent CN.)

Iodates and Ferric Salts. — The 1 : 20 aqueous solution, freshly prepared with recently boiled and cooled water, should not within one minute acquire a blue color on the addition of starch solution and 2 or 3 drops of diluted sulphuric acid. (Indicating less than 0.001 per cent I_2O_5, or less than 0.05 per cent Fe'''.)

Nitrates. — On heating 1 gm. of potassium iodide with 5 c.c. of sodium hydroxide solution and a mixture of 0.5 gm. of zinc dust and 0.5 gm. of powdered iron, no vapors of ammonia should be evolved (to be ascertained by moist litmus paper). (Indicating less than 0.0016 per cent N_2O_5.)

Quantitative Determination, and Test for Excess of Potassium Chloride.* — Dissolve 0.5 gm. of potassium iodide in 25 c.c. of water, add 35 c.c. of decinormal solution of silver nitrate, and shake until the precipitate has coagulated. Add 2 c.c. of nitric acid and 5 c.c. of cold, saturated solution of ferric ammonium sulphate, and titrate with decinormal solution of ammonium sulphocyanate. Not less than 4.7 c.c. and not more than 4.9 c.c. of the latter should be required to produce a permanent red color. (Indicating at most 0.5 per cent KCl.)

1 c.c. of decinormal $AgNO_3 = 0.016602$ gm. of KI, log. 22016.

POTASSIUM IODIDE, NEUTRAL

TESTS OF PURITY

Neutrality. — Dissolve 10 gm. of the neutral potassium iodide in 50 c.c. of water, in a stoppered, flint-glass bottle, overlay with 30 c.c. of ether, and add 3 drops of iodeosin solution. After vigorously shaking, the aqueous layer will exhibit a pale-red color which disappears on adding 1 drop of decinormal hydrochloric acid solution and thoroughly shaking. (Indicating less than 0.003 per cent alkali as KOH.)

* Potassium iodide nearly always contains potassium chloride. The amount of the latter present when this test is complied with, is at most 0.5 per cent.

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Should the water which has been used for making the solution of potassium iodide have an alkaline reaction to iodeosin, it must be first rendered neutral to it.

Further Tests. — The tests as given under Potassium Iodide are also to be made, and in the manner there described.

POTASSIUM NITRATE

KNO_3. Mol. Wt. 101.11

Colorless, transparent, prismatic crystals, or crystalline powder, permanent in air, and soluble in 4 parts of cold, and in about 0.5 part of boiling, water; almost insoluble in alcohol. The aqueous solution is neutral to litmus paper.

TESTS OF PURITY

Sulphates. — Dissolve 3 gm. of potassium nitrate in 60 c.c. of water, and add 1 c.c. of hydrochloric acid followed by barium chloride solution. No precipitate of barium sulphate should form on standing fifteen hours. (Indicating less than 0.0025 per cent SO_3.)

Chlorides. — Dissolve 1 gm. of potassium nitrate in 20 c.c. of water, acidulate with 1 c.c. of nitric acid and add silver nitrate solution. No change should occur. (Indicating less than 0.0005 per cent Cl.)

Chlorates and Perchlorates. — Gently ignite 1 gm. of potassium nitrate, dissolve the melt in 20 c.c. of water, add 1 c.c. of nitric acid and some silver nitrate solution. No change should appear. (Indicating less than 0.0005 per cent as Cl.)

Calcium and Heavy Metals. — The solution of 3 gm. of potassium nitrate in 50 c.c. of water should not be affected either by hydrogen sulphide water or by ammonia water followed by ammonium oxalate solution (indicating less than 0.01 per cent Ca); and after the addition of ammonium sulphide solution neither a green or brown color, nor a precipitate, should appear. (Indicating no heavy metals present.)

Iron. — 20 c.c. of the 1 : 20 aqueous solution, acidulated with 1 c.c. of hydrochloric acid should not be reddened by potassium sulphocyanate solution. (Indicating less than 0.0008 per cent Fe.)

Nitrites. — Dissolve 1 gm. of potassium nitrate in 20 c.c. of water, acidulate with 1 c.c. of 16 per cent sulphuric acid, and add 1 c.c. of a freshly prepared, colorless 1 : 200 solution of metaphenylenediamine hydrochloride.* No yellow or yellowish-brown color should develop. (Indicating less than 0.00055 per cent N_2O_3.)

POTASSIUM NITRITE

KNO_2. Mol. Wt. 85.11

White, or slightly yellowish, tough, hygroscopic, sticks, which are very easily soluble in water. The aqueous solution is alkaline to litmus paper. The preparation contains at least 90 per cent of KNO_2.

* Should the solution of metaphenylenediamine hydrochloride have a color, it is to be decolorized before using by warming with freshly ignited animal charcoal.

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TESTS OF PURITY

Heavy Metals. — Dissolve 1 gm. of potassium nitrite and 1 gm. of ammonium chloride in 5 c.c. of water, evaporate to dryness on the water-bath, and dissolve the residue in 10 c.c. of water. This solution should not be affected by hydrogen sulphide water; and after adding ammonia water and ammonium sulphide solution neither a green or brown color nor a precipitate should appear. (Indicating none present.)

Chlorides. — The solution of 1 gm. of potassium nitrite in 20 c.c. of water should acquire at most a slight opalescence on the addition of 5 c.c. of nitric acid and silver nitrate solution. (Indicating less than 0.002 per cent Cl.)

Sulphates. — On adding to 20 c.c. of the 1 : 20 aqueous solution 5 c.c. of concentrated nitric acid, boiling to expel oxides of nitrogen, and then adding barium nitrate solution, no turbidity should develop. (Indicating less than 0.175 per cent SO_3.)

Quantitative Determination. — Dissolve 1 gm. of potassium nitrite in water and dilute to 100 c.c. Allow 20 c.c. of the nitrite solution to run slowly into a mixture of 50 c.c. of decinormal potassium permanganate solution, 300 c.c. of water and 25 c.c. of 16 per cent sulphuric acid, while constantly shaking. After ten minutes add 3 gm. of potassium iodide to the mixture, and titrate the liberated iodine with decinormal sodium thiosulphate solution, using starch solution as indicator.

1 c.c. of decinormal $KMnO_4 = 0.0042555$ gm. of KNO_2, log. 62895.

POTASSIUM OXALATE, NEUTRAL

$K_2C_2O_4 + H_2O$. Mol. Wt. 184.22

Rhombic prisms, soluble in 3 parts of water.

TESTS OF PURITY

Neutrality. — The solution of 10 gm. of potassium oxalate in 100 c.c. of water should not be changed by phenolphthalein solution (indicating no alkali present); but on adding further 0.1 c.c. of decinormal potassium hydroxide solution, a red color should appear. (Indicating less than 0.0045 per cent acid as $H_2C_2O_4$.)

Sulphates. — Boil a solution of 5 gm. of potassium oxalate in 200 c.c. of water, and add 10 c.c. of hydrochloric acid followed by barium chloride solution. No precipitate of barium sulphate should form on standing fifteen hours. (Indicating less than 0.005 per cent SO_3.)

Chlorides. — Dissolve 1 gm. of potassium oxalate in 25 c.c. of water, and add 10 c.c. of nitric acid and two or three drops of silver nitrate solution. At most a slight opalescence should develop on shaking. (Indicating less than 0.0025 per cent Cl.)

Heavy Metals. — To a solution of 1 gm. of potassium oxalate in 25 c.c. of water, add hydrogen sulphide water. The solution should remain unchanged. On now adding ammonia water and a few drops of ammonium sulphide solution to the liquid, no green or brown color should develop, nor should a precipitate form. (Indicating none present.)

Quantitative Determination. — Dissolve 1 gm. of potassium oxalate in water and dilute to 100 c.c. To 25 c.c. of this solution add 6 to 8 c.c. of concentrated sulphuric acid, heat the liquid to about 60° C., and titrate with decinormal potassium permanganate solution.

1 c.c. of decinormal $KMnO_4 = 0.009211$ gm. of $K_2C_2O_4 + H_2O$, log. 96431.

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POTASSIUM PERCHLORATE

$KClO_4$. Mol. Wt. 138.56

Colorless, rhombic prisms, soluble in about 65 parts of cold, and in 8 parts of boiling, water, and insoluble in alcohol. Potassium perchlorate is neither colored by concentrated sulphuric acid nor decomposed by hydrochloric acid (difference from potassium chlorate.)

TESTS OF PURITY

Chlorides. — 20 c.c. of the 1 : 20 solution in hot water should develop at most a slight opalescence on the addition of silver nitrate solution. (Indicating less than 0.002 per cent Cl.)

Heavy Metals. — 20 c.c. of the 1 : 20 solution in hot water should not be affected by hydrogen sulphide water. After adding a few drops of ammonium sulphide solution neither a green or brown color nor a precipitate should appear. (Indicating none present.)

Nitrates. — On heating a mixture of 1 gm. of potassium perchlorate with 5 c.c. of sodium hydroxide solution (sp.gr. 1.3) and a mixture of 0.5 gm. of zinc dust and 0.5 gm. of powdered iron, no vapors of ammonia should be evolved (to be ascertained by moist litmus paper.) (Indicating less than 0.0016 per cent N_2O_5.)

Sulphates. — On adding barium chloride solution to 20 c.c. of the 1 : 20 solution in hot water, no change should appear. (Indicating less than 0.005 per cent SO_3.)

Calcium. — On adding ammonium oxalate solution to 20 c.c. of the 1 : 20 solution of potassium perchlorate in hot water, no change should occur. (Indicating less than 0.02 per cent Ca.)

Quantitative Determination. — Fuse in a nickel crucible a mixture of 1 gm. of powdered potassium perchlorate and 5 gm. of powdered sodium nitrite free from chlorine, and maintain in a state of fusion for about half an hour at a low temperature. When cool, dissolve the melt in water and dilute to 500 c.c. Add to 100 c.c. of this solution 20 c.c. of decinormal silver nitrate solution and 20 c.c. of nitric acid, and warm the mixture on the water-bath until the red vapors have disappeared. Now add 5 c.c. of a cold saturated solution of ferric ammonium sulphate and titrate with decinormal solution of ammonium sulphocyanate.

1 c.c. of decinormal $AgNO_3 = 0.013856$ gm. of $KClO_4$, log. 14163.

POTASSIUM PERMANGANATE

$KMnO_4$. Mol. Wt. 158.03

I

POTASSIUM PERMANGANATE

Dark-violet, almost black prisms, exhibiting a steel-blue luster; soluble in 16 parts of cold, and in 3 parts of boiling, water. The 1 : 1000 aqueous solution is neutral to litmus paper.

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TESTS OF PURITY

Sulphates and Chlorides. — Boil 0.5 gm. of potassium permanganate with a mixture of 2 c.c. of 95 per cent alcohol and 25 c.c. of water, and filter. The filtrate should be colorless, and, after adding to it 2 c.c. of nitric acid, neither barium nitrate nor silver nitrate solutions should cause more than a slight opalescence. (Indicating less than 0.03 per cent SO_3, and less than 0.004 per cent Cl.)

Nitrates. — Add gradually 1 gm. of crystallized oxalic acid to a solution of 0.5 gm. of potassium permanganate in 5 c.c. of water heated to 50° to 60° C., then heat to boiling, and filter. On overlaying 1 c.c. of ferrous sulphate solution on a cooled mixture of 2 c.c. of the clear colorless filtrate and 2 c.c. of concentrated sulphuric acid, no dark-colored zone should form at the contact-surfaces of the two liquids. (Indicating less than 0.08 per cent N_2O_5.)

Chlorates. — Heat 2 gm. of potassium permanganate in a platinum crucible, and gradually add small pieces of paraffin until no more glowing is observed. When cold, the residue is treated with 20 c.c. of water, filtered, and 5 c.c. of nitric acid and silver nitrate solution are added to the colorless filtrate. The liquid should develop at most a slight opalescence. (Indicating less than 0.0016 per cent Cl_2O_5.)

Quantitative Determination. — Dissolve 1 gm. of potassium permanganate in water and dilute to 100 c.c. Dilute 10 c.c. of this solution with 100 c.c. of water, add 2 gm. of potassium iodide and 20 c.c. of diluted sulphuric acid, and titrate with decinormal solution of sodium thiosulphate.

1 c.c. of decinormal $Na_2S_2O_3 = 0.0031606$ gm. of $KMnO_4$, log. 49977.

II

POTASSIUM PERMANGANATE, FREE FROM SULPHATES

The preparation contains about 100 per cent of $KMnO_4$.

TESTS OF PURITY

Sulphates. — The solution of 3 gm. of potassium permanganate in 150 c.c. of water with 15 c.c. of 95 per cent alcohol (or a little hydrogen peroxide, 30 per cent), heated, if necessary, until perfectly decolorized, should yield a filtrate, which, on the addition of 2 c.c. of hydrochloric acid and barium chloride solution, should afford no precipitate of barium sulphate on standing fifteen hours. (Indicating less than 0.003 per cent SO_3.)

Further Tests. — The other tests and the quantitative determination as given under Potassium Permanganate are also to be made.

POTASSIUM PERSULPHATE

$K_2S_2O_8$. Mol. Wt. 270.34

Colorless crystals, soluble in 50 parts of cold, and 4 parts of boiling, water. The aqueous solution is neutral to litmus paper. Potassium persulphate gradually decomposes, evolving oxygen. The preparation contains at least 95 per cent of $K_2S_2O_8$.

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TESTS OF PURITY

Chlorides. — The solution of 1 gm. of potassium persulphate in 50 c.c. of water should become at most slightly opalescent on adding 3 to 5 drops of silver nitrate solution. More silver nitrate solution produces a black precipitate which masks the chloride reaction. (Indicating less than 0.002 per cent Cl.)

Heavy Metals. — Dissolve 4 gm. of potassium persulphate in 50 c.c. of sulphurous acid (sp.gr. 1.029 to 1.035) with the aid of heat, evaporate the solution to dryness on the water-bath, and dissolve the residue in 40 c.c. of water.

20 c.c. of this solution should not be affected by hydrogen sulphide water. (Indicating none present.)

Another portion of 20 c.c. of the solution should not appear changed on adding ammonia water to alkaline reaction; and on the further addition of a few drops of ammonium sulphide solution, at most a green color may develop, but no precipitate should form. (Indicating at most a trace present.)

Quantitative Determination. — Weigh into a 100 c.c. graduated flask 1 gm. powdered potassium persulphate, pour over the sample a solution of 5 gm. of potassium iodide in 50 c.c. of water, add 10 c.c. of diluted sulphuric acid, let the mixture stand about a half-hour with frequent shaking, fill with water to the mark and mix. Titrate 20 c.c. of the solution with decinormal sodium thiosulphate solution.

1 c.c. of decinormal $Na_2S_2O_3 = 0.013517$ gm. of $K_2S_2O_8$, log. 13088.

POTASSIUM STANNOSULPHATE

(Marignac's Salt)

$K_2Sn(SO_4)_2$. Mol. Wt. 389.34

White crystals, soluble in dilute potassium or sodium hydroxide solution, and in hydrochloric acid.

TEST OF PURITY

Quantitative Determination. — Dissolve 1 gm. of potassium stannosulphate, 5 gm. of sodium bicarbonate, and 5 gm. of potassium and sodium tartrate in water, dilute to 100 c.c., and titrate 50 c.c. of the solution with decinormal iodine solution, using starch solution as the indicator.

1 c.c. of decinormal $I = 0.019467$ gm. of $K_2Sn(SO_4)_2$, log. 28930.

POTASSIUM SULPHATE

K_2SO_4. Mol. Wt. 174.36

White, hard crystals, soluble in 10 parts of cold, and in 4 parts of boiling, water, but insoluble in alcohol. The aqueous solution is neutral to litmus paper.

TESTS OF PURITY

Chlorides, Heavy Metals (Fe, Cu), Calcium and Magnesium. — 20 c.c. portions of the 1 : 20 aqueous solution should not be affected by hydrogen sulphide water (indicating no heavy metals present), or by ammonium oxalate, silver

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nitrate, or ammonum phosphate solutions. (Indicating less than 0.001 per cent Cl, less than 0.02 per cent Ca, and less than 0.005 per cent Mg.)

Iron. — The solution of 1 gm. of potassium sulphate in 20 c.c. of water boiled with a few drops of nitric acid should remain colorless on the addition of potassium sulphocyanate solution. (Indicating less than 0.0008 per cent Fe.)

Nitrates. — Dissolve 1 gm. of potassium sulphate and a small crystal of sodium chloride in 10 c.c. of water, add 1 drop of 1 : 1000 indigo solution and 10 c.c. of concentrated sulphuric acid. The blue color of the fluid should not disappear on shaking. (Indicating less than 0.0032 per cent N_2O_5.)

Ammonium Salts. — Dissolve 1 gm. of potassium sulphate in 20 c.c. of water and add a few drops of Nessler's solution. At most a slight yellow coloration should result. (Indicating less than 0.005 per cent NH_3).

POTASSIUM SULPHIDE

Leather-brown or yellowish-green pieces, which deliquesce in moist air, dissolve in 2 parts of water with but a slight residue, and afford alkaline, yellowish-green solutions. On adding acetic acid to the aqueous solution, hydrogen sulphide gas is copiously evolved.

TEST OF PURITY

Nitrogen. — Dissolve 5 gm. of potassium sulphide in 100 c.c. of water in a distillation flask, add 2 gm. of Devarda's metal and 50 c.c. of sodium hydroxide solution (nitrogen-free, sp.gr. 1.3), connect the flask with a bulb-tube, condenser, and a receiver containing 2 to 3 c.c. of fifth-normal solution of hydrochloric acid. After one to two hours distil off about 50 c.c. and titrate the distillate with fifth-normal solution of potassium hydroxide, using methyl orange as indicator. The amount of fifth-normal acid solution found to have been neutralized by the ammonia should not be more than 0.2 c.c. (Indicating not more than 0.0112 per cent N.)

POTASSIUM SULPHIDE SOLUTION

The solution contains 5 per cent of K_2S, and is intended for use in determining nitrogen according to Kjeldahl.

TEST OF PURITY

Nitrogen. — Mix 100 c.c. of the potassium sulphide solution in a distilling flask with 2 gm. of Devarda's metal and 50 c.c. of nitrogen-free sodium hydroxide solution (sp.gr. 1.3) and connect the flask with a bulb-tube, condenser, and a receiver containing 10 c.c. of water and 2 to 3 c.c. of fifth-normal solution of hydrochloric acid. After one or two hours distil off about 50 c.c. and titrate the distillate with normal solution of potassium hydroxide, using methyl orange as the indicator. The ammonia distilled over should not have neutralized more than 0.2 c.c. of the fifth-normal acid solution. (Indicating not more than 0.00056 per cent N.)

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POTASSIUM SULPHOCYANATE

(Potassium Thiocyanate)

KSCN. Mol. Wt. 97.18.

Colorless, prismatic crystals, deliquescent in the air, and easily soluble in water and in alcohol. The 1 : 20 aqueous solution is neutral to litmus paper.

TESTS OF PURITY

Substances Insoluble in Alcohol. — 1 gm. of potassium sulphocyanate should completely dissolve in 10 c.c. of boiling absolute alcohol, and yield a clear solution. (Indicating none present.)

Chlorides. — Dissolve 1 gm. of potassium sulphocyanate in 100 c.c. of water, add 25 c.c. of nitric acid, heat on the water-bath for three hours, and then add silver nitrate solution. At most a slight opalescence should appear. (Indicating less than 0.0025 per cent Cl.)

Sulphates. — On adding to a solution of 1 gm. of potassium sulphocyanate in 20 c.c. of water, a few drops of hydrochloric acid and some barium chloride solution, no turbidity should be observed within five minutes. (Indicating less than 0.01 per cent SO_3.)

Heavy Metals. — On dissolving 1 gm. of potassium sulphocyanate in 20 c.c. of water and adding hydrogen sulphide water, no change should appear; and on adding to the solution a few drops of ammonium sulphide solution, no precipitate should form, nor should a green or brown color develop. (Indicating none present.)

Iron. — The solution of 1 gm. of potassium sulphocyanate in 20 c.c. of water should remain perfectly colorless on the addition of 0.5 c.c. of hydrochloric acid. (Indicating less than 0.0004 per cent Fe.)

Ammonium Compounds. — On heating to boiling a solution of 3 gm. of potassium sulphocyanate in 10 c.c. of sodium hydroxide solution (sp.gr. 1.3), no ammonia should be evolved (to be ascertained by means of moistened litmus paper). (Indicating less than 0.0011 per cent NH_3.)

POTASSIUM SULPHYDRATE

(Potassium Hydrosulphide)

$2KSH+H_2O$. Mol. Wt. 162.37

Colorless, deliquescent crystals* easily soluble in water and in alcohol. The solution is strongly alkaline to litmus paper.

TEST OF PURITY

Polysulphides. — The solution of 1 gm. of potassium sulphydrate in 20 c.c. of water should be colorless and clear. On acidulating the solution with

* When exposed to the air, the preparation rapidly acquires a yellow color by taking up oxygen with the formation of polysuphide. It should, therefore, be preserved in well-stoppered bottles.

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hydrochloric acid, hydrogen sulphide gas is generated copiously, and the fluid exhibits a slight opalescent turbidity. No separation of sulphur should occur. (Indicating none present.)

POTASSIUM TETRAOXALATE

$KHC_2O_4+H_2C_2O_4+2H_2O$. Mol. Wt. 254.16

Colorless, triclinic crystals, soluble in 55 parts of cold, and in 3 parts of boiling, water. The aqueous solution is acid to litmus paper.

TESTS OF PURITY

Chlorides. — Dissolve 1 gm. of potassium tetraoxalate in 25 c.c. of water and 10 c.c. of nitric acid, and add 2 or 3 drops of silver nitrate solution. After shaking, the solution should be at most slightly opalescent. (Indicating less than 0.0025 per cent Cl.)

Sulphates. — Dissolve 4 gm. of potassium tetraoxalate in 200 c.c. of water, add 10 c.c. of hydrochloric acid, heat to boiling, and add barium chloride solution. No precipitate of barium sulphate should form on standing fifteen hours. (Indicating less than 0.0063 per cent SO_3.)

Heavy Metals. — The solution of 1 gm. of potassium tetraoxalate in 25 c.c. of warm water should not be affected by hydrogen sulphide water; and after adding 5 c.c. of ammonia water and a few drops of ammonium sulphide solution, neither a green or brown color should develop, nor should a precipitate form. (Indicating none present.)

Quantitative Determination. (*a*) Acidimetric: Dissolve 1 gm. of potassium tetraoxalate in water and dilute to 100 c.c. Titrate 50 c.c. of this solution, while boiling, with fifth-normal potassium hydroxide solution, using phenolphthalein as the indicator.

1 c.c. of fifth normal KOH = 0.016944 gm. of $KHC_2O_4+H_2C_2O_4+2H_2O$, log. 22901.

(*b*) Oxidimetric: To 25 c.c. of the solution made for test (*a*), add 6 to 8 c.c. of concentrated sulphuric acid, heat the liquid to about 60° C., and titrate with decinormal potassium permanganate solution.

1 c.c. of decinormal $KMnO_4$ = 0.006354 gm. of $KHC_2O_4+H_2C_2O_4+2H_2O$, log. 80305.

POTASSIUM AND SODIUM TARTRATE

(Rochelle Salt; Seignette Salt)

$KNaC_4H_4O_6+4H_2O$. Mol. Wt. 282.20

Colorless, transparent, prismatic crystals, soluble in 1.4 parts of water, and yielding a solution neutral to phenolphthalein.

TESTS OF PURITY

Calcium. — On dissolving 1 gm. of potassium and sodium tartrate in 10 c.c. of water, and adding to the solution 5 c.c. of diluted acetic acid, and then shaking for a few minutes, a white, crystalline precipitate forms. On filtering off the latter, diluting the filtrate with an equal volume of water, and then adding 8 to 10 drops of ammonium oxalate solution, no turbidity should develop within one minute. (Indicating less than 0.03 per cent Ca.)

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Heavy Metals. — 20 c.c. of the 1 : 20 aqueous solution should show no change with hydrogen sulphide water; and after adding a few drops of ammonium sulphide solution, neither a green or brown color nor a precipitate should appear. (Indicating none present.)

Chlorides and Sulphates. — The solution of 1 gm. of potassium and sodium tartrate in 10 c.c. of water, with 5 c.c. of nitric acid added, should not be affected by silver nitrate and barium nitrate solutions. (Indicating less than 0.0005 per cent Cl and less than 0.175 per cent SO_3.)

Ammonium Compounds. — On heating the solution of 1 gm. of potassium and sodium tartrate in 10 c.c. of water with 10 c.c. of sodium hydroxide solution (sp.gr. 1.3), no vapors of ammonia should be given off (to be ascertained by means of moist litmus paper). (Indicating less than 0.0045 per cent NH_3.)

PUMICE STONE, PLATINIZED

Fragments of pumice stone impregnated with platinum chloride solution and subsequently ignited. The preparation is used as a catalysing agent.

Dark-gray, irregular pieces about the size of peas, possessing a dull metallic luster.

PYROGALLOL

(Acid Pyrogallic)

$C_6H_3(OH)_3$. Mol. Wt. 126.05

White, shining needles or scales, melting at 131° C. Pyrogallol is soluble in 1.7 parts of water, in 1 part of alcohol, and in 1.5 parts of ether; it is difficultly soluble in benzene, chloroform, and carbon disulphide. The aqueous solution is slightly acid to litmus paper.

TESTS OF PURITY

Inorganic Matter. — 1 gm. of pyrogallol, on being heated, should volatilize and leave no weighable residue. (Indicating less than 0.05 per cent.)

Gallic Acid. — 2 gm. of pyrogallol must completely dissolve in 5 c.c. of ether (sp.gr. 0.72), and afford a clear solution. (Indicating less than 1 per cent gallic acid, $C_7H_6O_5$.)

QUARTZ SAND

This sand is prepared for laboratory use by washing and subsequently igniting it.

TESTS OF PURITY

Substances Soluble in Hydrochloric Acid. — Digest 20 gm. of quartz sand with a mixture of 20 c.c. of hydrochloric acid and 100 c.c. of water for about four hours on the water-bath with frequent shaking, filter, evaporate 50 c.c. of the filtrate to dryness and ignite the residue. The weight of the ignited residue should not exceed 0.001 gm. (Indicating not more than 0.03 per cent.)

Chlorides. — On shaking 1 gm. of quartz sand with 20 c.c. of water, filtering,

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and adding silver nitrate solution to the filtrate, at most a slight opalescence should appear. (Indicating less than 0.002 per cent Cl.)

Volatile Substances. — On igniting 5 gm. of quartz sand the loss in weight should not exceed 0.001 gm. (Indicating not more than 0.02 per cent.)

RESORCINOL

(Resorcin)

$C_6H_4(OH)_2$. **Mol. Wt. 110.05**

Colorless crystals, having a scarcely perceptible but peculiar odor, soluble in about 1 part of water, or alcohol, easily soluble in ether and in glycerin; difficultly soluble in chloroform and in carbon disulphide. Resorcinol volatilizes on being heated, and melts at 110° to 111° C. The aqueous solution is acid to litmus paper.

TESTS OF PURITY

Non-Volatile Matter. — 1 gm. of resorcinol, on being heated and volatilized, should leave no weighable residue. (Indicating less than 0.05 per cent.)

Diresorcin and Phenol. — 1 gm. of resorcinol should yield a perfectly clear solution with 20 c.c. of water; and the solution on being warmed should not have a phenolic odor. (Indicating none present.)

Free Acids (e.g., Salicylic Acid). — On dissolving 1 gm. of resorcinol in 10 c.c. of 85 per cent alcohol, and adding 1 drop of lacmoid solution, a wine-red liquid is obtained, which, on the addition of 1 drop of decinormal solution of potassium hydroxide, should assume a blue color. (Indicating none present.

SEA SAND

This sand is prepared for laboratory use by treatment with acid and subsequent ignition.

TESTS OF PURITY

Substances Soluble in Hydrochloric Acid. — Digest 20 gm. of sea-sand with a mixture of 20 c.c. of hydrochloric acid and 100 c.c. of water for about four hours on the water-bath with frequent shaking, filter, evaporate 50 c.c. of the filtrate, and ignite the residue. The weight of the ignited residue should not exceed 0.01 gm. (Indicating not more than 0.3 per cent.)

Chloride. — On shaking 1 gm. of sea sand with 20 c.c. of water, filtering, and adding silver nitrate solution to the filtrate, at most a slight opalescence should appear. (Indicating less than 0.002 per cent Cl.)

Volatile Substances. — On igniting 5 gm. of sea sand the loss in weight should not exceed 0.001 gm. (Indicating not more than 0.02 per cent.)

SILVER

Ag. Atomic Wt. 107.88

A white, shining metal, almost entirely insoluble in hydrochloric acid and in cold dilute sulphuric acid; soluble in nitric and in hot concentrated sulphuric acids.

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TESTS OF PURITY

Foreign Metals. — Dissolve 2 gm. of silver in 10 c.c. of nitric acid. The solution should be colorless and there should be no insoluble residue. Upon dilution with water no turbidity should occur. Precipitate the silver by adding hydrochloric acid to the boiling solution, filter, and evaporate the filtrate to dryness. No weighable residue should remain. (Indicating less than 0.025 per cent.)

Quantitative Determination. — 0.2 to 0.3 gm. of silver are dissolved in 10 c.c. of nitric acid with the aid of heat, continuing the heating until the red fumes have disappeared. After diluting with 100 c.c. of water and adding 5 c.c. of a cold, saturated solution of ammonium ferric sulphate, titrate with decinormal ammonium sulphocyanate solution.

1 c.c. of decinormal $NH_4SCN = 0.010788$ gm. of Ag, log. 03294.

SILVER NITRATE

$AgNO_3$. Mol. Wt. 169.89

Colorless, lustrous crystals or white sticks, exhibiting a stellate, crystalline fracture, and yielding a clear, colorless solution with 0.6 part of water, and with 14 parts of alcohol (about 85 per cent by weight). The aqueous solution should be neutral to litmus paper. The preparation contains 100 per cent of $AgNO_3$.

TESTS OF PURITY

Chlorides. — Dissolve 5 gm. of silver nitrate in 5 c.c. of water, and allow the solution to run into 100 c.c. of water. No turbidity or opalescence should appear. (Indicating less than 0.002 per cent Cl).

Potassium Nitrate. — Dissolve 0.5 gm. of silver nitrate in 0.5 c.c. of water, mix the solution with 20 c.c. of absolute alcohol, and shake for a few minutes. No turbidity or precipitate should form. (Indicating less than 2 per cent KNO_3.)

Salts of Copper, Bismuth, and Lead. — Dissolve 1 gm. of silver nitrate in 5 c.c. of water, and add to the solution 10 c.c. of ammonia water. The liquid should remain clear and colorless. (Indicating less than 0.002 per cent Cu, less than 0.02 per cent Bi, or less than 0.3 per cent Pb.)

Substances Not Precipitated by Hydrochloric Acid. — Dissolve 2 gm. of silver nitrate in 50 c.c. of water, heat the solution to boiling, and add 3 c.c. of hydrochloric acid. After the precipitate has settled, filter, and evaporate the filtrate to dryness. No weighable residue should remain. (Indicating less than 0.025 per cent.)

Quantitative Determination. — 1 gm. of silver nitrate is dissolved in water, and diluted to 100 c.c. 20 c.c. of this solution are mixed with 100 c.c. of water, 10 c.c. of nitric acid, and 5 c.c. of a cold, saturated solution of ammonium ferric sulphate, and then titrated with decinormal ammonium sulphocyanate solution.

1 c.c. of decinormal $NH_4SCN = 0.016989$ gm. of $AgNO_3$, log. 23016.

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SILVER NITRITE

$AgNO_2$. Mol. Wt. 153.89

Small, yellowish, acicular crystals, slowly soluble in about 300 parts of cold water. Silver nitrite is more readily soluble in hot water, but suffers partial decomposition therein.

TESTS OF PURITY

Substances Not Precipitated by Hydrochloric Acid. — Dissolve 2 gm. of silver nitrite, with heat, in 100 c.c. of water, with the aid of 2 c.c. of nitric acid, heat the solution to boiling, and add 3 c.c. of hydrochloric acid. After the precipitate has settled, filter, and evaporate the filtrate to dryness. No weighable residue should remain. (Indicating less than 0.025 per cent.)

Quantitative Determination. — Dissolve 1 gm. of silver nitrite in cold water, and dilute to 500 c.c. Make a mixture containing 10 c.c. of decinormal potassium permanganate solution, 10 c.c. of diluted sulphuric acid, and 300 c.c. of water, heat it to 40 to 50° C., and run the nitrite solution into it until the pink color just disappears. Care must be taken towards the end to introduce the nitrite solution very slowly, because the change from pink to colorless always requires some time.

1 c.c. of decinormal $KMnO_4$ = 0.0076945 gm. of $AgNO_2$, log. 88618.

SODIUM

Na. Atomic Wt. 23.00.

The metal, when freshly cut, has a silver-white surface, which rapidly becomes dull on exposure to air and becomes covered with crusts of sodium oxide, sodium hydroxide, and sodium carbonate. At ordinary temperatures, sodium has the consistency of wax, but at low temperatures it is brittle.

TESTS OF PURITY

Nitrogen Compounds and Foreign Metals. — (*a*) 1 gm. of sodium is freed from adhering petroleum by wiping with pieces of filtering paper, then cut into small pieces, and thrown upon 20 c.c. of cold water. The solution of sodium hydroxide so obtained should not have the odor of ammonia on warming (indicating less than 0.07 per cent N), and should appear unchanged upon adding ammonium sulphide solution. (Indicating no heavy metals present.)

(*b*) The solution prepared from 1 gm. of sodium and 20 c.c. of water should be unchanged in appearance on the addition of 10 c.c. of hydrochloric acid, followed by hydrogen sulphide water. (Indicating no heavy metals present.)

SODIUM ACETATE

$NaC_2H_3O_2+3H_2O$. Mol. Wt. 136.07

Colorless, transparent crystals, efflorescent in warm air, soluble in about 1 part of water, in 29 parts of cold, and 1 part of boiling, alcohol (about 85 per cent by weight).

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The solution of 1 gm. of sodium acetate in 1 c.c. of water is alkaline to litmus paper. It should, however, not be reddened, or only very slightly, by phenolphthalein solution; and any red color should disappear on adding 1 drop of decinormal hydrochloric acid solution.

TESTS OF PURITY

Chlorides. — The solution of 1 gm. of sodium acetate in 20 c.c. of water should appear unchanged on adding 1 c.c. of nitric acid followed by silver nitrate solution. (Indicating less than 0.0005 per cent Cl.)

Sulphates. — 20 c.c. of the 1 : 20 aqueous solution should not be rendered turbid by barium chloride solution. (Indicating less than 0.01 per cent SO_3.)

Heavy Metals and Calcium. — 20 c.c. of the 1 : 20 aqueous solution should not be affected by hydrogen sulphide water (indicating no heavy metals present), or by ammonium oxalate solution. (Indicating less than 0.02 per cent Ca.)

Iron. — The solution of 1 gm. of sodium acetate in 20 c.c. of water should not be reddened on adding 1 c.c. of hydrochloric acid, followed by potassium sulphocyanate solution. (Indicating less than 0.0008 per cent Fe.)

SODIUM AMALGAM

Gray pieces, the size of peas, containing 2 per cent of metallic sodium.

TEST OF STRENGTH

Quantitative Determination of Sodium. — Introduce 10 gm. of sodium amalgam into 100 c.c. of water, and allow the mixture to stand with repeated shaking until the evolution of hydrogen entirely ceases. Then titrate with normal solution of hydrochloric acid, using methyl orange as the indicator.

1 c.c. of normal HCl = 0.0230 gm. of Na, log. 36173.

SODIUM BICARBONATE

$NaHCO_3$. Mol. Wt. 84.01

White, crystalline crusts, or crystalline powder, soluble in 12 parts of water, but insoluble in alcohol. The aqueous solution is faintly alkaline to litmus paper.

TESTS OF PURITY

Sulphates. — Dissolve 2 gm. of sodium bicarbonate in 30 c.c. of water, add 10 c.c. of hydrochloric acid, heat to boiling, and add barium chloride solution. No precipitate of barium sulphate should form within fifteen hours. (Indicating less than 0.0125 per cent SO_3.)

Silicates. — Dissolve 5 gm. of sodium bicarbonate in 15 c.c. of water and 25 c.c. of hydrochloric acid in a platinum dish. Evaporate the solution on the water-bath, dry the residue for half an hour at 120° C., and then dissolve it in 3 c.c. of hydrochloric acid and 25 c.c. of water. The solution should be perfectly clear. (Indicating none present.)

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Chlorides and Thiosulphates. — On adding to the solution of 1 gm. of sodium bicarbonate in 47 c.c. of water, 3 c.c. of nitric acid, the solution should be clear; and on adding silver nitrate solution, at most a faint opalescence should develop within ten minutes. (Indicating less than 0.001 per cent Cl, and less than 0.0065 per cent $Na_2S_2O_3$.)

Phosphates. — Dissolve 2 gm. of sodium bicarbonate in 20 c.c. of water, add 20 c.c. of nitric acid and 10 c.c. of ammonium molybdate solution. On heating to 30° to 40° C., no yellow precipitate should form within two hours. (Indicating less than 0.033 per cent P_2O_5.)

Heavy Metals. — (*a*) The solution of 3 gm. of sodium bicarbonate in 40 c.c. of water and 8 c.c. of hydrochloric acid should remain unchanged on the addition of hydrogen sulphide water; on adding to the liquid 5 c.c. of ammonia water and a few drops of ammonium sulphide solution, no precipitate should form, nor should a green or brown color develop. (Indicating none present.)

(*b*) The solution of 1 gm. of sodium bicarbonate in 15 c.c. of water and 2 c.c. of hydrochloric acid after adding a few drops of nitric acid and boiling should not be more than slightly reddened on the addition of potassium sulphocyanate solution. (Indicating less than 0.0015 per cent Fe.)

Potassium. — The yellow color imparted by sodium bicarbonate to the flame, when observed through cobalt glass, should have at most a transient violet tinge. (Indicating less than 0.4 per cent K.)

Monocarbonate (Neutral Sodium Carbonate.) — (*a*) A solution of 1 gm. of sodium bicarbonate in 20 c.c. of water, prepared at a temperature not above 15° C., without excessive shaking, should not be more than slightly reddened on the addition of 3 drops of phenolphthalein solution. (Indicating at most a trace.)

(*b*) 1 gm. of the sodium bicarbonate dried over sulphuric acid before weighing, and ignited, should leave a residue weighing not more than 0.638 gm. (Indicating less than 2 per cent Na_2CO_3.)

Ammonium Compounds. — On heating 1 gm. of sodium bicarbonate in a test-tube, no vapors of ammonia should be evolved (to be ascertained by moistened turmeric paper). (Indicating less than 0.0032 per cent NH_3.)

Sulphocyanates. — The solution of 1 gm. of sodium bicarbonate in 3 c.c. of nitric acid and 47 c.c. of water should not be reddened on the addition of 1 drop of ferric chloride solution. (Indicating less than 0.1 per cent CNS.)

Quantitative Determination. — On titrating 1 gm. of sodium bicarbonate with normal solution of hydrochloric acid, using methyl orange as the indicator, 11.9 c.c. of the acid should be required.

1 c.c. of normal HCl = 0.08401 gm. of $NaHCO_3$, log. 92433.

SODIUM BISULPHATE

(Sodium Acid Sulphate)

$NaHSO_4+H_2O$. Mol. Wt. 138.09

Colorless crystals, easily soluble in water. The aqueous solution is strongly acid to litmus paper.

TESTS OF PURITY

Heavy Metals. — The solution of 1 gm. of sodium bisulphate in 20 c.c. of water should not be affected by hydrogen sulphide water; nor should a precipitate or a green or brown color be produced on making it alkaline with ammonia water, and then adding a few drops of ammonium sulphide solution. (Indicating none present.)

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Chlorides. — The 1 : 20 aqueous solution should not be rendered more than slightly opalescent on adding silver nitrate solution. (Indicating less than 0.002 per cent Cl).

Arsenic. — The mixture of 1 gm. of powdered sodium bisulphate with 3 c.c. of stannous chloride solution should not acquire a darker color within one hour. (Indicating less than 0.0015 per cent As.)

Potassium. — The yellow-colored flame produced by sodium bisulphate, when observed through cobalt glass, should exhibit only a transient violet color. (Indicating less than 0.4 per cent K.)

Quantitative Determination. — Dissolve 1 gm. of sodium bisulphate in 50 c.c. of water, and titrate with normal potassium hydroxide solution, using methyl orange as the indicator.

1 c.c. of normal KOH = 0.13809 gm. of $NaHSO_4+H_2O$, log. 14016.

SODIUM BISULPHITE

(Sodium Acid Sulphite)

$NaHSO_3$. Mol. Wt. 104.08.

A white powder, having an odor of sulphurous acid, and soluble in 4 parts of water. The aqueous solution is acid to litmus paper.

TESTS OF PURITY

Chlorides. — Dissolve 1 gm. of sodium bisulphite in 10 c.c. of water, add 10 c.c. of perhydrol (hydrogen peroxide, 30 per cent) and a solution of potassium hydroxide (purest) until the solution has an alkaline reaction. Evaporate the solution on the water-bath, dissolve the residue in 20 c.c. of water, acidulate with nitric acid, and add silver nitrate solution. At most a slight opalescence should appear. (Indicating less than 0.002 per cent Cl.)

Heavy Metals. — Dissolve 2 gm. of sodium bisulphite in 10 c.c. of water, add 20 c.c. of hydrochloric acid, and evaporate on the water-bath. Dissolve the residue in 20 c.c. of water and add to the solution hydrogen sulphide water no change should occur, and no precipitate should form on the further addition of ammonia water and ammonium sulphide solution. (Indicating none present.)

Arsenic. — Add 5 gm. of sodium bisulphite in small portions to 25 c.c. of nitric acid (sp.gr. 1.3), and afterwards add 5 c.c. of concentrated sulphuric acid and evaporate the mixture, first as far as possible on the water-bath, and finally on a sand-bath until fumes of sulphuric acid begin to be evolved. After cooling, dissolve the residue in 50 c.c. of water.

Set in operation a Marsh apparatus by means of 20 gm. of arsenic-free zinc and diluted sulphuric acid (1 : 5), and add the solution, prepared as above, in small portions to the generating flask of the apparatus. No deposit of arsenic should be visible in the reduction tube within one hour. (Indicating less than 0.0002 per cent As.)

Quantitative Determination. — Dissolve 1 gm. of sodium bisulphite in thoroughly boiled water, and dilute to 100 c.c. Run this solution from a burette into a mixture of 30 c.c. of decinormal iodine solution and 5 c.c. of hydrochloric acid, with constant shaking, until complete decoloration ensues.

1 c.c. of decinormal I = 0.005204 gm. of $NaHSO_3$, log. 71634.

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SODIUM BORATE, CRYSTALS

(Borax; Sodium Tetraborate; Sodium Biborate)

$Na_2B_4O_7+10H_2O$. Mol. Wt. 382.16

Hard, white crystals, or crystalline pieces, soluble in about 25 parts of cold, and in 0.5 part of boiling water, freely soluble in glycerin, but almost insoluble in alcohol. When borax is heated, it swells up, the water of crystallization being expelled, and at a red heat the anhydrous borax fuses to a transparent, colorless mass.

The aqueous solution is alkaline to litmus paper, and when acidulated with hydrochloric acid it colors turmeric paper brown. The brown color becomes particularly evident as the paper dries, and when moistened with ammonia water the color becomes greenish-black.

I

SODIUM BORATE

TESTS OF PURITY

Carbonates and Sulphates. — The solution of 1 gm. of borax in 20 c.c. of water should not effervesce on the addition of 5 c.c. of hydrochloric acid (indicating less than 2 per cent CO_2), and should not be affected by the subsequent addition of barium chloride solution. (Indicating less than 0.0875 per cent SO_2).

Chlorides. — On adding to 20 c.c. of the 1 : 20 aqueous solution 3 c.c. of nitric acid followed by silver nitrate solution, at most a slight opalescent turbidity should develop. (Indicating less than 0.002 per cent Cl.)

Quantitative Determination.* — Dissolve 1 gm. of borax in 50 c.c. of water, add 1 drop of methyl orange solution, and titrate with fifth-normal hydrochloric acid solution.

1 c.c. of fifth-normal HCl = 0.038216 gm. of $Na_2B_4O_7+10H_2O$, log. 58224.

II

SODIUM BORATE, PUREST, CRYSTALS

TESTS OF PURITY

Content of Water. †— 1 gm. of borax, on ignition, should yield a residue weighing 0.529 gm. (52.9 per cent.)

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* Compare method of Schwartz as given in Sutton, Volumet. Anal., 10 ed., p. 95 (1911).

† Besides this prismatic borax containing 10 molecules of water of crystallization, there is another borax containing 5 molecules of water of crystallization, crystallizing in octahedra.

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Carbonates and Sulphates. — The solution of 1 gm. of borax in 20 c.c. of water should not effervesce on adding 5 c.c. of hydrochloric acid, (indicating less than 2 per cent CO_2), nor should it become turbid on the subsequent addition of barium chloride solution. (Indicating less than 0.0875 per cent SO_3.)

Chlorides. — On adding to 20 c.c. of the 1 : 20 aqueous solution 3 c.c. of nitric acid. followed by silver nitrate solution, no change should appear. (Indicating less than 0.0005 per cent Cl.)

Calcium. — The solution of 1 gm. of borax in 20 c.c. of water should not be rendered turbid by ammonium oxalate solution. (Indicating less than 0.02 per cent Ca.)

Iron and Other Metals.—(*a*) The solution of 1 gm. of borax in 20 c.c. of water should not be reddened on the addition of 2 c.c. of hydrochloric acid, followed by potassium sulphocyanate solution. (Indicating less than 0.0008 per cent Fe.)

(*b*) The 1 : 20 aqueous solution, acidulated with 2 c.c. of hydrochloric acid, should remain unchanged on the addition of hydrogen sulphide water. (Indicating no other heavy metals present.)

Quantitative Determination. — The determination is carried out as detailed under Sodium Borate.

SODIUM BORATE, PUREST, CALCINED

A white, spongy mass, or white powder, containing at least 75 per cent of $Na_2B_4O_7$ (anhydrous).

TESTS OF PURITY

The tests to be made are those given under Sodium Borate, Purest, Crystals. But for each gram of crystallized borax use 0.7 gm. of the calcined.

Quantitative Determination.* — Dissolve 1 gm. of calcined borax in 50 c.c. of water, add 1 drop of methyl orange solution, and titrate with fifth-normal hydrochloric acid solution. At least 37.2 c.c. of the acid should be necessary to produce the end-point.

1 c.c. of fifth-normal HCl = 0.0202 gm. of $Na_2B_4O_7$, log. 30535.

SODIUM BORATE, PUREST, FUSED

(Borax Glass; Anhydrous Borax)

$Na_2B_4O_7$. Mol. Wt. 202.0

Colorless, vitreous pieces, which take up moisture from the air, and in consequence become opaque.

TESTS OF PURITY

The tests to be made are those given under Sodium Borate, Purest, Crystals. But for each gram of the crystallized borax use 0.5 gm. of the fused. Pulverize the pieces in an agate mortar.

Quantitative Determination. — This is carried out as described under Sodium Borate, Purest, Calcined.

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* Compare methods given by Sutton, Volumet. Anal., 10 ed., p. 95 (1911).

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SODIUM BROMATE

$NaBrO_3$ Mol. Wt. 150.92

Lustrous crystals, or crystalline powder, soluble in 3 parts of cold, and in about 1.5 parts of boiling, water.

TESTS OF PURITY

Bromides. — On dissolving 2 gm. of sodium bromate in 20 c.c. of water, and adding diluted sulphuric acid, the solution should not immediately acquire a yellow color. (Indicating less than 0.03 per cent Br.)

Quantitative Determination. — Dissolve 0.10 to 0.15 gm. of sodium bromate in 20 c.c. of water, add 3 gm. of potassium iodide and 5 c.c. of hydrochloric acid, and titrate the liberated iodine with decinormal sodium thiosulphate solution.

1 c.c. of decinormal $Na_2S_2O_3 = 0.0025153$ gm. of $NaBrO_3$, log. 40059.

SODIUM CARBONATE

I

SODIUM CARBONATE, CRYSTALS

$Na_2CO_3 + 10H_2O$. Mol. Wt. 285.16

Colorless, transparent crystals, efflorescent in the air, soluble in 1.6 parts of cold, and 0.2 part of boiling, water. The aqueous solution is strongly alkaline to litmus paper. Sodium carbonate is insoluble in absolute alcohol. The crystallized sodium carbonate contains 37 per cent of the anhydrous salt, Na_2CO_3.

TESTS OF PURITY

Substances Insoluble in Water. — 20 gm. of sodium carbonate should completely dissolve in 80 c.c. of water, yielding a perfectly colorless solution. (Indicating none present.)

Sodium Hydroxide. — In a graduated flask of 100 c.c. capacity, dissolve 3 gm. of crystallized sodium carbonate in 50 c.c. of water, add to the solution 6 gm. of crystallized barium chloride dissolved in 30 c.c. of water, and then fill with water up to the mark. After thoroughly shaking, filter, and to 50 c.c. of the filtrate add some phenolphthalein solution. The liquid should not acquire a red color. Use boiled water throughout. (Indicating less than 0.1 per cent NaOH.)

Sulphates. — On boiling a solution of 10 gm. of sodium carbonate in 50 c.c. of water and 10 c.c. of hydrochloric acid, for a few minutes, and then adding barium chloride solution, no precipitate of barium sulphate should form within fifteen hours. (Indicating less than 0.00125 per cent SO_3.)

Chlorides. — The solution of 5 gm. of sodium carbonate in 50 c.c. of water and 10 c.c. of nitric acid should not be affected by silver nitrate solution. (Indicating less than 0.0001 per cent Cl.)

Silicates. — Dissolve 20 gm. of crystallized sodium carbonate in 30 c.c. of hydrochloric acid in a platinum dish, and evaporate the solution on the water-

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bath to dryness. Dry the residue half an hour at 120° C., and then dissolve it in 3 c.c. of hydrochloric acid and 50 c.c. of water. This solution should be perfectly clear. (Indicating none present.)

Nitrates. — Dissolve 1 gm. of sodium carbonate in 10 c.c. of diluted sulphuric acid, and overlay this liquid on 5 c.c. of a solution of 0.5 gm. of diphenylamine in 100 c.c. of concentrated sulphuric acid and 20 c.c. of water. No blue color should form at the contact-surfaces of the two liquids. (Indicating less than 0.007 per cent N_2O_5.)

Phosphates. — To the solution of 20 gm. of sodium carbonate in 50 c.c. of nitric acid add 50 c.c. of a solution of ammonium molybdate in nitric acid. No yellow precipitate should form in the liquid on standing two to three hours at about 40° C. (Indicating less than 0.001 per cent P_2O_5.)

Potassium. — The yellow color imparted by sodium carbonate to the flame, when observed through cobalt glass, should at most have a transient violet tinge. (Indicating less than 0.4 per cent K.)

Ammonium Compounds. — On adding 1 c.c. of Nessler's reagent to a solution of 10 gm. of sodium carbonate in 50 c.c. of water, no change should be observed. (Indicating less than 0.0001 per cent NH_3.)

Calcium and Magnesium. — Dissolve 10 gm. of sodium carbonate in 10 c.c. of water and 10 c.c. of hydrochloric acid, and add 5 c.c. of ammonia water, followed by ammonium oxalate solution. The liquid should not become turbid (indicating less than 0.002 per cent Ca), and no precipitate should form on the addition of ammonium phosphate solution and standing for several hours. (Indicating less than 0.0002 per cent Mg.)

Heavy Metals. — (*a*) The solution of 20 gm. of sodium carbonate in 50 c.c. of water and 20 c.c. of hydrochloric acid should not be affected by hydrogen sulphide water; on now adding to the solution 5 c.c. of ammonia water and a few drops of ammonium sulphide solution, no turbidity or green color should develop. (Indicating none present.)

(*b*) A solution of 10 gm. of sodium carbonate in 25 c.c. of water and 10 c.c. of hydrochloric acid should not acquire a red color on the addition of potassium sulphocyanate solution. (Indicating less than 0.00008 per cent Fe.)

Arsenic. — Introduce 20 gm. of arsenic-free, metallic zinc into the generating flask of a Marsh apparatus, and start the hydrogen by adding dilute sulphuric acid (1 : 5). Dissolve 30 gm. of sodium carbonate in 100 c.c. of dilute sulphuric acid (1 : 5), and introduce the solution in small quantities at a time into the Marsh apparatus. After one hour no deposit of arsenic should be visible within the reduction tube. (Indicating less than 0.00003 per cent As.)

Quantitative Determination. — Dissolve 3 gm. of sodium carbonate in 50 c.c. of water, and titrate with normal solution of hydrochloric acid, using methyl orange as the indicator.

1 c.c. of normal HCl = 0.14308 gm. of $Na_2CO_3+10H_2O$, log. 15558.

II

SODIUM CARBONATE, ANHYDROUS

Na_2CO_3. Mol. Wt. 106.0

A white powder, containing 99 to 100 per cent of Na_2CO_3.*

* Sodium carbonate, which is to be used as a standard in volumetric determinations, must first be heated for thirty minutes on a sand-bath, or in an air-bath, at 270° to 300° C. See Lunge, Z. angew. Chem., **17**, 231 (1904); *abst.*, J. Chem. Soc., **86**, II, 289 (1904).

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TESTS OF PURITY

The tests to be made are those given under Sodium Carbonate Crystals. But for each gram of crystals use 0.35 gm. of the anhydrous.

Quantitative Determination. — Dissolve 1 gm. in 50 c.c. of water and titrate with normal solution of hydrochloric acid, using methyl orange as indicator. At least 18.7 c.c. of the normal acid should be required.

1 c.c. of normal HCl = 0.0530 gm. of Na_2CO_3, log. 72428.

SODIUM CHLORIDE

NaCl. Mol. Wt. 58.46

I

SODIUM CHLORIDE

White, cubical crystals, or crystalline powder, soluble in 2.9 parts of water. The aqueous solution is neutral to litmus paper.

TESTS OF PURITY

Sulphates. — 3 gm. of sodium chloride yield a perfectly clear solution with 20 c.c. of water. On diluting this solution with 80 c.c. of water, adding 1 c.c. of hydrochloric acid, heating to boiling, and then adding barium chloride solution, no precipitate of barium sulphate should form within fifteen hours. (Indicating less than 0.0066 per cent SO_3.)

Alkaline Earths and Heavy Metals. — The solution of 3 gm. of sodium chloride in 50 c.c. of water, heated to boiling, should not be affected by ammonium oxalate solution; nor by sodium carbonate solution; nor by ammonium sulphide solution. (Indicating no heavy metals, and less than 0.01 per cent earths as Ca.)

Magnesium. — Dissolve 3 gm. of sodium chloride in 10 c.c. of water, and add 5 c.c. of ammonia water and ammonium phosphate solution. No precipitate should form within three hours. (Indicating less than 0.0007 per cent Mg.)

Iodides. — On adding to 20 c.c. of the 1 : 20 aqueous solution, one drop of ferric chloride solution and some starch solution, no blue color should develop. (Indicating less than 0.15 per cent I.)

Potassium. — On adding 5 c.c. of sodium cobaltic nitrite solution to the solution of 1 gm. of sodium chloride in 10 c.c. of water, no precipitate should form within fifteen hours. (Indicating less than 0.18 per cent K.)

Iron. — The solution of 3 gm. of sodium chloride in 20 c.c. of water boiled with a few drops of nitric acid should not acquire a red color on the addition of potassium sulphocyanate solution. (Indicating less than 0.0003 per cent Fe.)

Ammonium. — The solution of 3 gm. of sodium chloride in 20 c.c. of water should not be changed in appearance on the addition of Nessler's reagent. (Indicating less than 0.0003 per cent NH_3.)

Quantitative Determination. — Dissolve 0.2 gm. of sodium chloride in 100 c.c. of water, add a few drops of potassium chromate solution, and titrate with decinormal silver nitrate solution.

1 c.c. of decinormal $AgNO_3$ = 0.005846 gm. of NaCl, log. 76686.

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II

SODIUM CHLORIDE, FUSED

Colorless or slightly reddish, translucent pieces.

TESTS OF PURITY

The tests and the quantitative determination as given under Sodium Chloride are to be made.

SODIUM HYDROXIDE

(Caustic Soda; Sodium Hydrate)

NaOH. Mol. Wt. 40.058

Three grades of sodium hydroxide are used in the chemical laboratory:

1. Sodium Hydroxide, from Sodium.
2. Sodium Hydroxide, Pure.
3. Sodium Hydroxide, Purified.

These three preparations vary chiefly in their content of chloride, sulphate, silicate and alumina.

I

SODIUM HYDROXIDE, FROM SODIUM

White, very hygroscopic pieces showing a crystalline structure on fracture. The preparation contains from 95 to 98 per cent of NaOH.

TESTS OF PURITY

Sulphates. — Dissolve 3 gm. of sodium hydroxide in 50 c.c. of water, acidulate with 15 c.c. of hydrochloric acid, heat to boiling, and add barium chloride solution. No precipitate of barium sulphate should form within fifteen hours. (Indicating less than 0.004 per cent SO_3.)

Chlorides. — Dissolve 1 gm. of sodium hydroxide in 20 c.c. of water, add 10 c.c. of nitric acid and a few drops of silver nitrate solution. At most a slight opalescent turbidity should develop. (Indicating less than 0.0025 per cent Cl.)

Nitrogen. —To 2 gm. of sodium hydroxide and a granule of sodium chloride dissolved in 10 c.c. of diluted sulphuric acid add 1 drop of 1 : 1000 indigo solution and 10 c.c. of concentrated sulphuric acid. The blue color of the mixture should not disappear on agitation. (Indicating less than 0.002 per cent N_2O_5.)

Nitrogen as Nitrates, Nitrites, Ammonia, etc. — Dissolve 50 gm. of sodium hydroxide in 100 c.c. of water in a distillation flask, and add 2 gm. of Devarda's metal. Connect the flask with a bulb tube, condenser, and a receiver containing 2 to 3 c.c. of fifth-normal hydrochloric acid solution and 10 c.c. of water, allow to stand one to two hours, and then distil off about 50 c.c. Titrate the

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distillate with fifth-normal potassium hydroxide solution, using methyl orange as the indicator. Not more than 0.2 c.c. of the acid solution should have been required to neutralize the ammonia. (Indicating less than 0.00112 per cent N.)

Nitrites. — The solution of 1 gm. of sodium hydroxide in 10 c.c. of water and 10 c.c. of diluted sulphuric acid should not acquire a blue color on the addition of zinc iodide-starch solution. (Indicating less than 0.00011 per cent N_2O_3.)

Phosphates. — Dissolve 5 gm. of sodium hydroxide in 50 c.c. of water and add 35 c.c. of nitric acid and 25 c.c. of a solution of ammonium molybdate in nitric acid. No yellow precipitate should form on standing two hours at about 40° C. (Indicating less than 0.003 per cent P_2O_5.)

Silicates. — Dissolve 5 gm. of sodium hydroxide in 25 c.c. of water and 25 c.c. of hydrochloric acid in a platinum dish, and evaporate to dryness on a water-bath. Dry the residue for half an hour at 120° C., and then dissolve it in 10 c.c. of hydrochloric acid and 90 c.c. of water. Any insoluble residue should be filtered off, washed, and ignited. Its weight should not exceed 0.001 gm. (Indicating not more than 0.02 per cent SiO_2.)

Aluminum, Calcium, and Heavy Metals.— Dissolve 50 gm. of sodium hydroxide in 250 c.c. of water, add 90 c.c. of acetic acid (sp.gr. 1.064), followed by 40 c.c. of ammonia water, and heat the liquid on the water-bath until the odor of ammonia has disappeared. Add a few drops of ammonia water, and allow to stand fifteen hours. Within this time, no precipitate, or only a very slight flocculent precipitate, should form. In the latter case, the precipitate is filtered off and washed. Its weight upon ignition should not exceed 0.001 gm. (Indicating not more than 0.00106 per cent Al.) With the filtrate brought to a volume of 400 c.c. the following tests are made:

To 20 c.c. diluted with 30 c.c. of water, add ammonium oxalate solution. No precipitate of calcium oxalate should form on standing two hours. (Indicating less than 0.012 per cent Ca.)

To another 20 c.c. add a few drops of ammonium sulphide solution; no precipitate should form, nor should a brown or green color appear. (Indicating no heavy metals.)

Quantitative Determination and Determination of Sodium Carbonate Content. — Titrate the solution of 1 gm. of sodium hydroxide in 100 c.c. of water with normal hydrochloric acid solution in the cold, using phenolphthalein as indicator. At least 24.1 c.c. of normal acid solution should be required to destroy the red color. Now add 1 drop of methyl orange, and titrate further until the color changes to red. In the second titration, at most 0.3 c.c. of the acid solution should be used. (Indicating not more than 3.18 per cent of Na_2CO_3.)*

1 c.c. of normal HCl = 0.04001 gm. of NaOH, log. 60217.

1 c.c. of normal HCl = 0.0530 gm. of Na_2CO_3, log. 72428.

II

SODIUM HYDROXIDE, PURE

White, very hygroscopic sticks or pieces, exhibiting a crystalline fracture. The preparation contains 95 to 98 per cent of NaOH.

TESTS OF PURITY

Sulphates. — Dissolve 3 gm. of sodium hydroxide in 50 c.c. of water, acidulate with 15 c.c. of hydrochloric acid, heat to boiling, and add barium chloride solution.

* Regarding the calculation of the sodium carbonate, see the note under Potassium Hydroxide, Purest.

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The liquid should not have more than a slight turbidity; and it should still be transparent when observed in a test-tube of 2 cm. diameter. (Indicating less than 0.025 per cent SO_3.)

Chlorides. — The solution of 1 gm. of sodium hydroxide in 20 c.c. of water and 10 c.c. of nitric acid may be rendered opalescent by silver nitrate solution, but should develop no precipitate at once. (Indicating less than 0.005 per cent Cl.)

Nitrates and Nitrogen as Nitrates, Nitrites, Ammonia, etc. — The tests are carried out as detailed under Sodium Hydroxide, from Sodium.

Silicates. — Dissolve 5 gm. of sodium hydroxide in 25 c.c. of water with 25 c.c. of hydrochloric acid in a platinum dish, and evaporate to dryness on the water-bath. Dry the residue for half an hour at 120° C., and then dissolve it in 10 c.c. of hydrochloric acid and 90 c.c. of water. Any insoluble residue is filtered off, washed, and ignited. Its weight after ignition should not exceed 0.003 gm. (Indicating not more than 0.06 per cent SiO_2.)

Aluminum, Calcium, and Heavy Metals. — 5 gm. of sodium hydroxide should afford a clear and colorless solution with 20 c.c. of water. Dilute the solution with 80 c.c. of water, add 30 c.c. of acetic acid (sp.gr. 1.041) and 10 c.c. of ammonia water, and heat upon the water-bath until the odor of ammonia has disappeared. After adding a few drops of ammonia water only a slight precipitate should form on standing fifteen hours. The precipitate is filtered off, washed, and ignited; its weight should then not exceed 0.003 gm. (indicating not over 0.032 per cent Al.) The filtrate should not be affected by ammonium oxalate solution (indicating less than 0.005 per cent Ca); and on adding ammonium sulphide solution, at most a slight green color may appear, in no case should a brown color or a precipitate be produced. (Indicating at most a trace of heavy metals.)

Quantitative Determination and Determination of Sodium Carbonate Content. — This is carried out as described under Sodium Hydroxide, from Sodium. The amount of sodium carbonate present should not exceed 4 per cent.

III

SODIUM HYDROXIDE, PURIFIED

White, very hygroscopic sticks or pieces, exhibiting a crystalline fracture. The preparation contains 90 to 95 per cent of NaOH.

TESTS OF PURITY

Nitrogen as Nitrates and Nitrites. — To the solution of 2 gm. of sodium hydroxide and a granule of sodium chloride in 10 c.c. of 16 per cent sulphuric acid add 1 drop of 1 : 1000 indigo solution and then 10 c.c. of concentrated sulphuric acid. The blue color of the mixture should not disappear on agitation. (Indicating less than 0.002 per cent N_2O_5.)

Aluminum, Calcium, and Heavy Metals. — 2.5 gm. of sodium hydroxide should afford a clear and colorless solution with 10 c.c. of water. Dilute the solution with 90 c.c. of water and add 15 c.c. of acetic acid (sp.gr. 1.041) and 5 c.c. of ammonia water. No precipitate should form. (Indicating less than 0.16 per cent Al.) The solution thus tested should not immediately be rendered turbid on the addition of ammonium oxalate solution (indicating less than 0.01 per cent Ca); and on adding to it ammonium sulphide solution, it should acquire at most a slight green color. (Indicating at most a trace of heavy metals.)

Quantitative Determination and Determination of the Sodium Carbonate Content. — Titrate a solution of 1 gm. of sodium hydroxide in 100 c.c. of water with normal hydrochloric acid solution in the cold, using phenolphthalein as the

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indicator. At least 23 c.c. of normal acid solution should be required to discharge the color. Now add 1 drop of methyl orange, and titrate further until the color again changes to red. In this second titration at most 0.5 c.c. of the acid solution should be necessary. (Indicating not more than 5.30 per cent of Na_2CO_3.)*

1 c.c. of normal HCl = 0.04001 gm. of NaOH, log. 60217.

1 c.c. of normal HCl = 0.0530 gm. of Na_2CO_3, log. 72428.

SODIUM HYDROXIDE SOLUTIONS

(Caustic Soda Solutions)

I

SODIUM HYDROXIDE, SOLUTION I—FREE FROM NITROGEN

A colorless liquid of specific gravity 1.3, and containing about 27 per cent of sodium hydroxide, NaOH.

TESTS OF PURITY

Nitrogen as Nitrates, Nitrites, Ammonia, etc. — Place 200 c.c. of the sodium hydroxide solution in a distillation flask, add 2 gm. of Devarda's metal, and connect the flask with a bulb tube, a condenser, and a receiver containing 2 to 3 c.c. of fifth-normal solution of hydrochloric acid and 10 c.c. of water. After the mixture has stood one to two hours, distil off about 70 c.c. and titrate the distillate with fifth-normal solution of potassium hydroxide, using methyl orange as the indicator. Not more than 0.2 c.c. of the fifth-normal hydrochloric acid solution should have been neutralized by the ammonia from the sodium hydroxide. (Indicating not more than 0.0002 per cent N.)

II

SODIUM HYDROXIDE, SOLUTION II—FREE FROM NITROGEN

A colorless liquid of specific gravity 1.3 and containing about 27 per cent of sodium hydroxide, NaOH.

TESTS OF PURITY

The test for nitrogen is to be made as described under Sodium Hydroxide, Solution I. The tests for Sulphates, Chlorides, Silicates, Aluminum, Calcium, and Heavy Metals are to be carried out as directed under Sodium Hydroxide, Pure. But for each gram of sodium hydroxide, use 2.9 c.c. (3.7 gm.) of sodium hydroxide, solution II.

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* Regarding the calculation of the sodium carbonate, see the note under Potassium Hydroxide, Purest.

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III

SODIUM HYDROXIDE, SOLUTION III. Sp.Gr. 1.168

A clear, colorless liquid, of specific gravity 1.168 to 1.172, and containing about 15 per cent of sodium hydroxide, NaOH.

TESTS OF PURITY

The tests to be made are those given under Sodium Hydroxide, Pure. But for each gram of sodium hydroxide, use 5.6 c.c. (6.5 gm.) of sodium hydroxide, solution III.

SODIUM HYDROXIDE WITH LIME

(Soda-Lime)

I

SODIUM HYDROXIDE WITH LIME

A white, porous mass, finely or coarsely granulated.

TESTS OF PURITY

Excess of Carbonate. — 5 gm. of soda-lime, when treated with dilute sulphuric acid, should not effervesce strongly. (Indicating less than 5 per cent CO_2.)

Nitrogen. — (*a*) On igniting soda-lime in a test-tube, it should develop no vapors of ammonia (to be ascertained by moistened litmus paper.) (Indicating no ammonia-nitrogen present.)

(*b*) To the solution of 50 gm. of soda-lime in 200 c.c. of hydrochloric acid add 2 gm. of Devarda's metal, and let the mixture stand, with frequent shaking, until the evolution of hydrogen has ceased. To the cold fluid add 100 c.c. of sodium hydroxide solution nitrogen-free (sp.gr. 1.3), and distil off about 100 c.c., collecting the distillate in a receiver containing 2 to 3 c.c. of fifth-normal hydrochloric acid solution and 10 c.c. of water. Titrate the distillate with fifth-normal potassium hydroxide solution, using methyl orange as the indicator. No more than 0.2 c.c. of the acid solution should have been required to neutralize the ammonia. (Indicating not more than 0.00112 per cent N.)

II

SODIUM HYDROXIDE WITH LIME FROM ICELAND SPAR

A white, porous, granular mass.

TESTS OF PURITY

Chlorides. — Dissolve 5 gm. of soda-lime in 50 c.c. of nitric acid, and dilute the solution with 100 c.c. of water. The liquid should acquire at most a slight opalescent turbidity on the addition of silver nitrate solution. (Indicating less than 0.001 per cent Cl.)

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Phosphates. — Dissolve 10 gm. of soda-lime in 100 c.c. of nitric acid, and add to the solution 25 c.c. of a solution of ammonium molybdate in nitric acid. No yellow precipitate should form within two to three hours on standing at about 40° C. (Indicating less than 0.003 per cent P_2O_5.)

Sulphur. — Thoroughly mix 5 gm. of finely powdered soda-lime with 2 gm. of sodium nitrate, and ignite the mixture in a silver crucible. Dissolve the melt in 50 c.c. of water with 20 c.c. of hydrochloric acid, filter, and to the filtrate add barium chloride solution. No precipitate of barium sulphate should form within fifteen hours. (Indicating less than 0.0015 per cent S.)

SODIUM INDIGOSULPHONATE

(Indigo Carmine)

$C_{16}H_8N_2O_2(SO_3Na)_2$. Mol. Wt. 466.22.

A dark blue powder, soluble in water.

TESTS OF PURITY

Water Content. — On drying sodium indigosulphonate at 100° C., the loss in weight should not exceed 10 per cent.

Determination of Indigo Content. —Dissolve 1 gm. of sodium indigosulphonate in water in a liter graduated flask, add 10 c.c. of concentrated sulphuric acid and fill to the mark with water. Dilute 100 c.c. of this solution with 600 c.c. of water and titrate in a porcelain dish with decinormal potassium permanganate solution. It is to be noted that the end-point of the titration is the color change from green to light yellow, and not the incipient red coloration.

1 c.c. of decinormal $KMnO_4$ = 0.007493 gm. of $C_{16}H_{10}N_2O_2$ (indigotin),* log. 87466.

SODIUM NITRATE

$NaNO_3$. Mol. Wt. 85.01

Colorless, transparent, rhombohedric crystals, soluble in 1.2 parts of water and in 80 parts of alcohol of 85 per cent by weight. The aqueous solution is neutral to litmus paper.

TESTS OF PURITY

Sulphates. — On dissolving 3 gm. of sodium nitrate in 60 c.c. of water, and adding 1 c.c. of hydrochloric acid followed by barium chloride solution, no precipitate of barium sulphate should form on standing fifteen hours. (Indicating less than 0.0025 per cent SO_3.)

Chlorides. — The solution of 1 gm. of sodium nitrate in 20 c.c. of water acidulated with 1 c.c. of nitric acid, should not be rendered turbid on the addition of silver nitrate solution. (Indicating less than 0.0015 per cent Cl.)

Chlorates and Perchlorates. — On gently igniting 1 gm. of sodium nitrate, dissolving the residue in 20 c.c. of water, and adding 1 c.c. of nitric acid, the

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* O. Miller and J. Smirnoff, Ber., **41**, 1366 (1908); *abst.*, J. Chem. Soc., **94**, I, 468 (1908).

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solution should not be rendered turbid on adding silver nitrate solution. (Indicating less than 0.005 per cent as Cl.)

Calcium and Heavy Metals. — The solution of 3 gm. of sodium nitrate in 50 c.c. of water should show no change either on the addition of hydrogen sulphide water or of ammonia water and ammonium oxalate solution. (Indicating less than 0.01 per cent Ca.) On adding ammonium sulphide solution neither a green or brown color nor a precipitate should appear. (Indicating no heavy metals present.)

Iron. — 20 c.c. of the 1 : 20 aqueous solution, acidulated with 1 c.c. of hydrochloric acid, should not be reddened on the addition of potassium sulphocyanate solution. (Indicating less than 0.0008 per cent Fe.)

Nitrites. — Dissolve 1 gm. of sodium nitrate in 20 c.c. of water, and add 1 c.c. of diluted sulphuric acid and 1 c.c. of a freshly prepared 1 : 200 colorless solution of metaphenylenediamine hydrochloride.* No yellow or yellowish-brown color should develop. (Indicating less than 0.00055 per cent N_2O_3.)

Iodates and Nitrites. — To 5 c.c. of the 1 : 20 aqueous solution add 3 or 4 drops of diluted sulphuric acid and zinc iodide-starch solution. No blue color should develop within one minute. (Indicating less than 0.004 per cent I_2O_5, and less than 0.00005 per cent N_2O_3.)

Potassium. — Dissolve 3 gm. of sodium nitrate in 6 c.c. of water with 3 to 5 drops of diluted acetic acid, and add 5 to 10 drops of solution of sodium-cobaltic nitrite. The liquid should remain clear, and on standing two hours at about 50° C., no yellow precipitate should form. (Indicating less than 0.33 per cent K.)

SODIUM NITRITE

$NaNO_2$. Mol. Wt. 69.01

I

SODIUM NITRITE

White, or slightly yellowish, tough sticks, becoming moist in the air, and soluble in about 2 parts of water. The aqueous solution is alkaline to litmus paper. The preparation contains at least 98 per cent of $NaNO_2$.

TESTS OF PURITY

Chlorides. — The solution of 1 gm. of sodium nitrite in 20 c.c. of water should not acquire more than a slight opalescent turbidity on the addition of 5 c.c. of nitric acid and some silver nitrate solution. (Indicating less than 0.002 per cent Cl.)

Sulphates. — The solution of 1 gm. of sodium nitrite in 20 c.c. of water, acidulated with 5 c.c. of nitric acid, should not be rendered turbid on the addition of barium nitrate solution. (Indicating less than 0.175 per cent SO_3.)

Heavy Metals. — Dissolve 1 gm. of sodium nitrite and 1 gm. of ammonium chloride in 5 c.c. of water, evaporate the solution to dryness on the water-bath, and dissolve the residue in 10 c.c. of water. This solution should not be affected by hydrogen sulphide water; and after the addition of ammonia water and ammonium sulphide solution no green or brown color should develop, nor should a precipitate form. (Indicating none present.)

* Should the solution of metaphenylenediamine hydrochloride have a color, it should be decolorized before use by warming with ignited animal charcoal.

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Quantitative Determination. — Dissolve 1 gm. of sodium nitrite in water and dilute to 200 c.c. Make a mixture of 50 c.c. of decinormal potassium permanganate solution with 300 c.c. of water and 25 c.c. of diluted sulphuric acid, and, while constantly shaking, allow 30 c.c. of the nitrite solution to run slowly into it. After ten minutes add to the solution 3 gm. of potassium iodide, and titrate the liberated iodine with decinormal sodium thiosulphate solution, using starch solution as the indicator.

1 c.c. of decinormal $KMnO_4 = 0.0034505$ gm. of $NaNO_2$, log. 53788.

II

SODIUM NITRITE, FREE FROM POTASSIUM

TESTS OF PURITY

Potassium. — To a solution of 5 gm. of crystallized cobalt acetate in 12 c.c. of water add a solution of 10 gm. of sodium nitrite in 20 c.c. of water previously acidulated with 2 c.c. of diluted acetic acid. Dilute the solution with 25 c.c. of water and allow it to stand twenty-four hours at 40° to 50° C. No yellow precipitate should form within this time. (Indicating less than 0.03 per cent K.)

Other Tests. — The other tests and the quantitative determination given under Sodium Nitrite should also be made, observing the conditions there described.

SODIUM NITROFERRICYANIDE

(Sodium Nitroprusside)

$Na_2Fe(CN)_5(NO) + 2H_2O$. Mol. Wt. 297.93

Ruby-red, transparent crystals, affording a clear solution with 2.5 parts of water.

TEST OF PURITY

Sulphate. — The solution of 1 gm. of sodium nitroprusside in 50 c.c. of water acidulated with 1 c.c. of hydrochloric acid should not be rendered turbid on adding barium chloride solution. (Indicating less than 0.01 per cent SO_3.)

SODIUM OXALATE

(Sörensen's Oxalate)

$Na_2C_2O_4$. Mol. Wt. 134.0

A white, crystalline powder, soluble in 31 parts of cold, and in 16 parts of boiling, water. Sodium oxalate, which is to be used in acidimetric and oxidimetric determinations as a standard, is obtained by precipitation with alcohol, and drying at 240° C. The preparation contains 100 per cent of $Na_2C_2O_4$.

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TESTS OF PURITY

Hygroscopic Moisture.* — 10 gm. of sodium oxalate, when dried in a water-drying-oven for twenty-four hours, should not lose more than 0.001 gm. in weight. (Indicating not more than 0.01 per cent.)

Sodium Carbonate, Sodium Binoxalate. — Introduce into a conical Jena flask about 250 c.c. of water and 10 drops of phenolphthalein solution (0.5 gm. phenolphthalein dissolved in 50 c.c. of alcohol and 50 c.c. of water), and evaporate to about 180 c.c. while passing in a current of pure air, free from carbon dioxide. Allow to cool to the ordinary temperature, add 5 gm. of sodium oxalate, shake carefully, and maintain the current of air. The oxalate slowly dissolves. If the solution is red, not more than 0.2 c.c. of decinormal acid solution should be required to render it colorless. But, if the solution is colorless, it must acquire a distinct red color on the addition of at most 0.1 c.c. of decinormal sodium hydroxide solution. (Indicating less than 0.0212 per cent, Na_2CO_3 or less than 0.0224 per cent $NaHC_2O_4$.)

Chlorides and Sulphates. — Decompose 10 gm. of sodium oxalate by heating in a platinum crucible, best over an alcohol lamp (illuminating gas contains sulphur). The carbonate formed is dissolved in nitric acid, and the solution filtered off from the carbon. On adding silver nitrate solution to half of the filtrate, no reaction for hydrochloric acid should be obtained (indicating less than 0.0001 per cent Cl); and in the other half no reaction for sulphuric acid should be obtained on adding barium nitrate solution. (Indicating less than 0.025 per cent SO_3.)

Iron and Potassium. — Decompose 10 gm. of sodium oxalate by igniting moderately in a platinum crucible, removing the last traces of carbon by cautious ignition with a blast lamp. The residue, when treated with warm water in a platinum dish, should be completely soluble, and should leave at most a scarcely weighable trace of undissolved iron oxide. The solution is filtered if necessary, and supersaturated with hydrochloric acid as free from iron as possible. It is then evaporated in a platinum dish on the water-bath, and the residue dried for two hours in the drying oven at 120° C. The residue must dissolve clear in water and the solution should give:

(*a*) With potassium sulphocyanate solution only a very faint reaction for iron after boiling with a little nitric acid (indicating less than 0.0003 per cent Fe) and

(*b*) With sodium and cobalt nitrite solution, no reaction for potassium. (Indicating less than 0.06 per cent K.)

Foreign Organic Bodies. — In a clean, thoroughly ignited test-tube, heat 1 gm. of sodium oxalate with 10 c.c. of pure concentrated sulphuric acid, as long as evolution of gas occurs, at first, gently, and then more strongly, until vapors of sulphuric acid begin to be evolved. When cooled, the color of the sulphuric acid is compared with that of another portion of 10 c.c. of sulphuric acid similarly treated, but without the addition of sodium oxalate. The sodium oxalate, treated in the manner described, should impart to the sulphuric acid not more than an exceedingly faint brownish tinge. (Indicating at most a trace present.)

Quantitative Determination. — 0.4 to 0.5 gm. of the sodium oxalate dried to constant weight at 100° C. are cautiously heated in a covered platinum crucible. In order to avoid the error due to the sulphur in illuminating gas, the crucible is held in a sheet of asbestos provided with a round hole, or a Berzelius alcohol lamp is used. The conversion of the oxalate into carbonate is completed in from fifteen to thirty minutes; any carbon present is then burned off by more strongly heating the crucible while half covered. When cold, dissolve the contents of

* These tests are taken from the original paper by S. P. L. Sörensen, Z. anal. Chem., 42, 512 (1903); *abst.*, J. Chem. Soc., 84, II, 684, 750 (1903).

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the crucible in water, and titrate in the cold with fifth-normal hydrochloric acid solution, using methyl orange as the indicator.

1 c.c. of fifth-normal HCl = 0.01340 gm. of $Na_2C_2O_4$, log. 12710.

Literature: S. P. L. Sörensen, Z. anal. Chem., **36**, 639 (1897); *abst.*, J. Chem. Soc., **74**, II, 185 (1898). Z. anal. Chem., **42**, 333 (1903); *abst.*, J. Chem. Soc., **84**, II, 684 (1903). Z. anal. Chem., **42**, 512 (1903); *abst.*, J. Chem. Soc., **84**, II, 684, 750 (1903); Z. anal. Chem., **44**, 156 (1905); *abst.*, J. Chem. Soc., **88**, II, 415 (1905). G. Lunge, Z. angew. Chem., **17**, 230, 269 (1904); *abst.*, J. Chem. Soc., **86**, II, 289 (1904). Sörensen, Z. anal. Chem., **45**, 217 (1906); *abst.*, J. Chem. Soc., **90**, II, 389 (1906). G. Lunge, Z. angew. Chem., **18**, 1520 (1905); *abst.*, J. Am. Chem. Soc., **28**, 1744 (1906). Sörensen and Andersen, Z. anal. Chem., **44**, 156 (1905); *abst.*, J. Chem. Soc., **88**, II, 415 (1905).

SODIUM PEROXIDE

(Sodium Superoxide)

Na_2O_2. Mol. Wt. 78.0

A light-yellow powder, very easily soluble in water with the copious evolution of oxygen and great elevation of temperature. On cautiously adding sodium peroxide to a cooled, dilute mineral acid, hydrogen peroxide results. The preparation contains at least 92 per cent of Na_2O_2.

TESTS OF PURITY

Sulphates. — Add 5 gm. of sodium peroxide in small quantities at a time to a mixture of 25 c.c. of hydrochloric acid and 100 c.c. of water. The clear solution should develop no precipitate with barium chloride solution on standing fifteen hours. (Indicating less than 0.014 per cent SO_3.)

Chlorides. — Add 3 gm. of sodium peroxide in small quantities at a time to a mixture of 20 c.c. of nitric acid and 100 c.c. of water. The clear liquid should exhibit at most a slight opalescent turbidity on the addition of silver nitrate solution. (Indicating less than 0.0015 per cent Cl.)

Phosphates. — Add 2.5 gm. of sodium peroxide in small quantities at a time to a mixture of 20 c.c. of nitric acid and 100 c.c. of water. On now adding 25 c.c. of a solution of ammonium molybdate in nitric acid and heating to 30° to 40° C., no yellow precipitate should form within two hours. (Indicating less than 0.006 per cent P_2O_5.)

Nitrogen. — Mix 1 gm. of sodium peroxide with 0.3 gm. of grape sugar, very cautiously, in a capacious nickel crucible. Effect the deflagration of the mixture by very gently heating the bottom of the covered crucible. When cold, dissolve the residue in 5 c.c. of water, acidulate with 10 c.c. of diluted sulphuric acid, and overlay 10 c.c. of this solution on 5 c.c. of diphenylamine solution (see Diphenylamine). No blue color should develop at the contact-surfaces of the two liquids. (Indicating less than 0.003 per cent N.)

Silicates. — Add 10 gm. of sodium peroxide in small portions to a mixture of 50 c.c. of hydrochloric acid and 100 c.c. of water, evaporate the solution to dryness on the water-bath, dry the residue half an hour at about 120° C., and dissolve it in 10 c.c. of hydrochloric acid and 90 c.c. of water. Any insoluble residue, when filtered off, washed and ignited, should weigh not more than 0.001 gm. (Indicating not more than 0.01 per cent SiO_2.)

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Heavy Metals. — On cautiously adding 5 gm. of sodium peroxide to 100 c.c. of water, a perfectly clear and almost colorless solution should result.

40 c.c. of this solution, acidulated with 10 c.c. of hydrochloric acid, should not be affected by hydrogen sulphide water. (Indicating none present.)

40 c.c. of the alkaline solution should develop no precipitate on the addition of a few drops of ammonium sulphide solution, nor should the liquid acquire a brown or a green color. (Indicating none present.)

Quantitative Determination. — The quantitative determination of sodium peroxide is most simply affected by decomposing it in a Lunge gas volumeter,* using cobalt nitrate as a catalyzer and measuring the volume of oxygen evolved.

Introduce 0.5 gm. of sodium peroxide in a weighing flask into the outer compartment of the decomposition flask, and in the inner place a mixture of 15 c.c. of diluted sulphuric acid and 2 or 3 drops of a saturated solution of cobalt nitrate. On tilting the flask, the liquid is allowed to mix with the sodium peroxide, whereby the decomposition of the latter is effected.

1 c.c. of O at 0° C. and 760 mm. = 0.006964 gm. of Na_2O_2, log. 84286.

SODIUM PHOSPHATE

(Disodium Hydrogen Phosphate; Secondary Sodium Phosphate)

$Na_2HPO_4 + 12H_2O$. Mol. Wt. 358.24

Colorless, transparent crystals, efflorescent in dry air, and soluble in 6 parts of water. The aqueous solution is alkaline to litmus paper.

TESTS OF PURITY

Carbonates and Sulphates. — 20 c.c. of the 1 : 20 aqueous solution should not effervesce on the addition of 1 c.c. of hydrochloric acid (indicating less than 2 per cent CO_2); nor should a precipitate of barium sulphate form within three hours on the subsequent addition of barium chloride solution. (Indicating less than 0.0075 per cent SO_3.)

Chlorides. — The solution of 1 gm. of sodium phosphate in 20 c.c. of water, acidulated with 2 c.c. of nitric acid, should show at most a very slight opalescent turbidity on the addition of silver nitrate solution. (Indicating less than 0.0015 per cent Cl.)

Nitrates. — To a solution of 2 gm. of sodium phosphate and a granule of sodium chloride in 10 c.c. of water add 5 c.c. of diluted sulphuric acid, 1 drop of 1 : 1000 indigo solution, and 10 c.c. of concentrated sulphuric acid. The blue color of the liquid should not disappear on agitation. (Indicating less than 0.0016 per cent N_2O_5.)

Heavy Metals. — The solution of 2 gm. of sodium phosphate in 20 c.c. of water, acidulated with 1 c.c. of hydrochloric acid, should appear unchanged on the addition of hydrogen sulphide water. On now adding to the liquid 5 c.c. of ammonia water and a few drops of ammonium sulphide solution, no precipitate should form, nor should a green color develop. (Indicating none present.)

Arsenic. — Into the generating flask of a Marsh apparatus introduce 10 gm. of arsenic-free, metallic zinc, and start the hydrogen with sulphuric acid (1 : 5).

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* Lunge, Chem.-Tech. Untersuch, 5 ed., **1**, 158 (1905); see also Olsen's Quantitative Analysis, 300 (1904).

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Dissolve 2 gm. of sodium phosphate in 50 c.c. of water and introduce the solution in small portions at a time into the Marsh apparatus. No deposit of arsenic should be visible in the reduction tube within one hour. (Indicating less than 0.0005 per cent As.)

Potassium. — The color imparted by sodium phosphate to a colorless flame, when observed through cobalt glass, should not be tinged with violet, or should be only momentarily so. (Indicating less than 0.4 per cent K.)

SODIUM PYROPHOSPHATE

$Na_4P_2O_7+10\ H_2O$. Mol. Wt. 446.24

Colorless, transparent crystals, soluble in 10 to 12 parts of cold water, and in somewhat more than 1 part of boiling water, but insoluble in alcohol. The aqueous solution is alkaline to litmus paper.

TESTS OF PURITY

Phosphates. — The 1 : 20 aqueous solution of sodium pyrophosphate should give a pure white precipitate with silver nitrate solution. (Indicating less than 2 per cent P_2O_5 as orthophosphate.)

Other Tests. — The tests as given under Sodium Phosphate are also to be made.

SODIUM SULPHATE

$Na_2SO_4+10H_2O$. Mol. Wt. 322.23

Colorless, efflorescent crystals, soluble in 3 parts of cold, and in 0.4 part of boiling, water, but insoluble in alcohol. The aqueous solution is neutral to litmus paper.

TESTS OF PURITY

Substances Insoluble in Water. — 5 gm. of sodium sulphate should afford a clear solution with 50 c.c. of water. (Indicating none present.)

Heavy Metals. — The solution of 1 gm. of sodium sulphate in 20 c.c. of water should not be affected by hydrogen sulphide water. (Indicating none present.)

The solution of 1 gm. of sodium sulphate in 20 c.c. of water and boiled with a few drops of nitric acid should not be reddened on the addition of potassium sulphocyanate solution. (Indicating less than 0.0008 per cent Fe.)

Chlorides. — The solution of 1 gm. of sodium sulphate in 20 c.c. of water should not be affected by silver nitrate solution. (Indicating less than 0.001 per cent Cl.)

Calcium and Magnesium. — The 1 : 20 aqueous solution should not be affected by ammonium oxalate solution (indicating less than 0.02 per cent Ca); nor, after adding ammonia water, by ammonium phosphate solution. (Indicating less than 0.005 per cent Mg.)

Arsenic. — Introduce 20 gm. of arsenic-free, metallic zinc into the generating flask of a Marsh apparatus, and start the hydrogen with diluted sulphuric acid (1 : 5). Dissolve 2 gm. of sodium sulphate in 20 c.c. of water and introduce the

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solution in small portions at a time into the Marsh apparatus. No deposit of arsenic should be visible in the reduction tube within one hour. (Indicating less than 0.0005 per cent As.)

SODIUM SULPHIDE

Na_2S+9H_2O. Mol. Wt. 240.2

Colorless, transparent crystals, easily and clearly soluble in water. The aqueous solution is alkaline to litmus paper. The preparation contains at least 97 per cent of Na_2S+9H_2O.

TESTS OF PURITY

Nitrogen. — To the solution of 10 gm. of sodium sulphide in 100 c.c. of water in a distillation flask add 2 gm. of Devarda's metal and 50 c.c. of nitrogen-free sodium hydroxide solution (sp.gr. 1.3), and connect the flask with a bulb tube, a condenser, and a receiver containing 2 to 3 c.c. of fifth-normal hydrochloric acid solution, and 10 c.c. of water. After allowing the mixture to stand one to two hours, distil off about 50 c.c. and titrate the distillate with fifth-normal potassium hydroxide solution, using methyl orange as indicator. The amount of fifth-normal hydrochloric acid solution found to have been neutralized by the ammonia should be at most 0.2 c.c. (Indicating not more than 0.0056 per cent N.)

Quantitative Determination; Sulphites, and Thiosulphates. — (*a*) Dissolve 1 gm. of sodium sulphide in water and dilute to 100 c.c. Allow 20 c.c. of this solution to run into a mixture of 20 c.c. of decinormal iodine solution, and 100 c.c. of water. After adding 3 c.c. of hydrochloric acid, titrate the excess of iodine with decinormal sodium thiosulphate solution, using starch solution as the indicator.

1 c.c. of decinormal I = 0.0120105 gm. of Na_2S+9H_2O, log. 07956.

(*b*) To a solution of 1 gm. of sodium sulphide in 100 c.c. of water, add 2 gm. of crystallized zinc sulphate dissolved in 150 c.c. of water; shake vigorously, allow to stand half an hour, and then filter. Titrate 50 c.c. of the filtrate with decinormal iodine solution, using starch solution as the indicator. Not more than 0.1 c.c. of decinormal iodine solution should be required. (Indicating not more than 0.08 per cent S as sulphites and thiosulphates.)

SODIUM SULPHIDE SOLUTION

Sodium sulphide solution contains 5 per cent of Na_2S and is used for determination of nitrogen according to Kjeldahl.

TEST OF PURITY

Nitrogen. — The test for nitrogen is to be carried out as described under Potassium Sulphide Solution.

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SODIUM SULPHITE

I

SODIUM SULPHITE, CRYSTALS

$Na_2SO_3+7H_2O$. Mol. Wt. 252.18

Colorless, prismatic crystals, efflorescent in air, and easily soluble in water. The aqueous solution is alkaline to litmus paper.

TESTS OF PURITY

Chlorides. — Dissolve 1 gm. of sodium sulphite in 10 c.c. of water, add 10 c.c. of perhydrol (hydrogen peroxide, 30 per cent) and a solution of potassium hydroxide, purest, to alkaline reaction, and evaporate the solution on the water-bath. Dissolve the residue in 20 c.c. of water, acidulate with nitric acid, and add silver nitrate solution. At most a slight opalescence should develop. (Indicating less than 0.002 per cent Cl.)

Heavy Metals. — Dissolve 2 gm. of sodium sulphite in 10 c.c. of water, add 20 c.c. of hydrochloric acid, evaporate on the water-bath, and dissolve the residue in 20 c.c. of water. On adding hydrogen sulphide water to this solution no change should occur; and after adding ammonia water and ammonium sulphide solution, no precipitate should form. (Indicating none present.)

Arsenic. — Introduce 5 gm. of sodium sulphite in small portions into 25 c.c. of nitric acid (sp.gr. 1.3). Thereupon add 5 c.c. of concentrated sulphuric acid, and evaporate the mixture on the water-bath as far as possible, and then on the sand-bath until sulphuric acid vapors begin to be evolved. Dissolve the residue in 50 c.c. of water.

Start the evolution of hydrogen in a Marsh apparatus by means of 20 gm. of granulated, arsenic-free zinc and diluted sulphuric acid (1 : 5), and introduce in small portions into the generating flask of the apparatus the solution to be tested. No deposit of arsenic should be visible in the reduction tube within one hour. (Indicating less than 0.0002 per cent As.)

Quantitative Determination. — Dissolve 1 gm. of sodium sulphite in boiled water and dilute to 100 c.c. Allow the solution to run from a burette into a mixture of 30 c.c. of decinormal iodine and 5 c.c. of hydrochloric acid, with constant agitation, until complete decolorization occurs.

1 c.c. of decinormal I = 0.012609 gm. of $Na_2SO_3+7H_2O$, log. 10068.

II

SODIUM SULPHITE, DRIED

A white powder, containing 85 to 90 per cent of Na_2SO_3.

TESTS OF PURITY

The tests for chlorides, heavy metals, and arsenic, as given under Sodium Sulphite, Crystals, are to be made; but for 1 gm. of the crystals use 0.5 gm. of the sodium sulphite, dried.

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The quantitative determination is to be made as described under Sodium Sulphite, Crystals.

1 c.c. of decinormal I = 0.0063035 gm. of Na_2SO_3, log. 79958.

SODIUM THIOSULPHATE

(Sodium Hyposulphite)

$Na_2S_2O_3+5H_2O$. Mol. Wt. 248.22

Colorless and odorless crystals, permanent in the air at ordinary temperatures. Sodium thiosulphate liquefies at 50° C. in its own water of crystallization, and is soluble in less than 1 part of cold water. The 1 : 1 aqueous solution is slightly alkaline to litmus paper. For making the tests the solutions are to be made with freshly boiled water.

TESTS OF PURITY

Carbonates, Sulphates, and Sulphites.* — To a solution of 3 gm. of sodium thiosulphate in 50 c.c. of water, add decinormal iodine solution (about 120 c.c.) until the liquid has a slight yellow color; on now adding barium chloride solution, no turbidity should ensue. (Indicating less than 0.17 per cent CO_2, and less than 0.01 per cent sulphates and sulphites as SO_3.)

Free Alkali. — The solution of 1 gm. of sodium thiosulphate in 10 c.c. of water should not be reddened by phenolphthalein solution. (Indicating less than 0.01 per cent as NaOH.)

Sulphides. — The solution of 1 gm. of sodium thiosulphate in 10 c.c. of water should not change in appearance on the addition of zinc sulphate solution. (Indicating less than 0.013 per cent S.)

Calcium. — The solution of 1 gm. of sodium thiosulphate in 20 c.c. of water should not be rendered turbid on the addition of ammonia water and ammonium oxalate solution. (Indicating less than 0.02 per cent Ca.)

Quantitative Determination. — Dissolve 1 gm. of sodium thiosulphate in 100 c.c. of water, and titrate with decinormal iodine solution, using starch solution as the indicator.

1 c.c. of decinormal I = 0.024822 gm. of $Na_2S_2O_3+5H_2O$, log. 39484.

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* Concerning a new titrimetric determination of thiosulphate in the presence of sulphite, see A. Gutman, Z. anal. Chem., **46**, 485 (1907); *abst.*, J. Chem. Soc., **92**, II, 812 (1907).

Regarding the determination of sulphite in the presence of thiosulphate, see G. Hübener, Chem. Ztg., **30**, 58 (1906); *abst.*, J. Chem. Soc., **90**, II, 196 (1906).

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SODIUM TUNGSTATE

(Sodium Wolframate)

$Na_2WO_4+2H_2O$.* **Mol. Wt. 330.03**

Colorless prisms or rhombic plates, soluble in 4 parts of water. The aqueous solution is alkaline to litmus paper.

TESTS OF PURITY

Water. — After gently igniting 1 gm. of sodium tungstate, the residue should weigh 0.88 gm. (Indicating 12 per cent water.)

Chlorides. — Dissolve 1 gm. of sodium tungstate in 20 c.c. of water, add 5 c.c. of nitric acid, and filter. The filtrate should acquire at most a slight opalescent turbidity within ten minutes after adding a few drops of silver nitrate solution. (Indicating less than 0.001 per cent Cl.)

Sulphates. — Dissolve 1 gm. of sodium tungstate in 25 c.c. of water, add 5 c.c. of nitric acid, boil for ten to fifteen minutes, and then filter. 20 c.c. of the filtrate should not immediately be rendered turbid on the addition of barium nitrate solution. (Indicating less than 0.075 per cent SO_3.)

Quantitative Determination. — Dissolve 1 gm. of sodium tungstate in 10 c.c. of water, add 10 c.c. of hydrochloric acid, evaporate on the water-bath to dryness, and heat the residue for half an hour at 120° C. Treat the residue with 20 c.c. of hydrochloric acid, and repeat the entire operation three or four times. Finally digest the residue with ammonium nitrate solution to which a little nitric acid has been added, filter, wash the tungstic acid with dilute nitric acid, dry, ignite, and weigh the residue. The latter should weigh at least 0.69 gm.

SODIUM AND AMMONIUM PHOSPHATE

(Microcosmic Salt)

$NH_4NaHPO_4+4H_2O$. **Mol. Wt. 209.15**

Colorless, monoclinic crystals, soluble in 5 parts of water. The aqueous solution is alkaline to litmus paper. Sodium and ammonium phosphate when fused on a platinum wire should yield a clear and colorless bead.

TESTS OF PURITY

The tests given under Sodium Phosphate are to be made, observing the conditions there described.

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* The preparation contains small amounts of sodium paratungstate ($Na_{10}W_{12}O_{41}+28H_2O$).

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SODIUM AND POTASSIUM CARBONATE, FUSED, ANHYDROUS

White sticks used for making carbon dioxide free from air, according to Kreussler.

SODIUM-COBALTIC NITRITE SOLUTION

A solution of 10 gm. of cobalt acetate in 25 c.c. of water is mixed with a solution of 20 gm. of sodium nitrite in 40 c.c. of water, and then acidulated with 5 c.c. of dilute acetic acid.

TETRAMETHYLPARAPHENYLENEDIAMINE HYDROCHLORIDE

(Paratetramethylphenylenediamine Hydrochloride)

$(CH_3)_2NC_6H_4N(CH_3)_2HCl$. Mol. Wt. 237.08

A white or slightly colored, crystalline powder, easily soluble in water, more difficultly in alcohol. It should be protected from light and moisture.

TESTS OF PURITY

Inorganic Matter. — On igniting 1 gm. of tetramethylparaphenylenediamine hydrochloride, no weighable residue should remain. (Indicating less than 0.05 per cent.)

Quantitative Determination. — Dissolve 0.5 gm. of tetramethylparaphenylenediamine hydrochloride in 50 c.c. of alcohol (85 per cent by weight) in a glass-stoppered flask, and titrate the solution with fifth-normal potassium hydroxide solution, using Poirrier's blue as the indicator.

1 c.c. of fifth-normal KOH = 0.023708 gm. of $(CH_3)_2NC_6H_4N(CH_3)_2HCl$, log. 37489.

NOTE. — Regarding the use of tetramethylparaphenylenediamine hydrochloride as a reagent, see Wurster., Ber., **19**, 3195 (1886); *abst.*, J. Chem. Soc., **52**, 295 (1887); Ber., **21**, 921 (1888); *abst.*, J. Chem. Soc., **54**, 627 (1888).

THYMOL

$C_6H_3(CH_3)(OH)(C_3H_7)1:3:4$. Mol. Wt. 150.11

Colorless, hexagonal crystals, easily soluble in alcohol, ether, and chloroform, but very difficultly soluble in water (1 : 1100). Thymol melts at 50° to 51° C., and boils at 228° to 230° C.

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TESTS OF PURITY

Inorganic Matter. — 1 gm. of thymol on heating should volatilize without leaving a weighable residue. (Indicating less than 0.05 per cent.)

Free Acids. — Blue litmus paper should not be reddened by the aqueous or alcoholic solution. (Indicating none present.)

Phenol. — The cold, saturated, aqueous solution acquires a milky turbidity on the addition of bromine water, but should show no crystalline precipitate. Nor should the aqueous solution of thymol afford a violet color with ferric chloride solution. (Indicating none present.)

TIN

Sn. Atomic Wt. 119

A soft, almost silver-white metal, melting at 231° C. Tin dissolves in hot hydrochloric acid with the formation of stannous chloride. Hot concentrated nitric acid converts it into insoluble meta-stannic acid.

TESTS OF PURITY

Lead, Copper, Iron, and Zinc. — Digest 5 gm. of the tin with 40 c.c. of nitric acid (sp.gr. 1.4) on the water-bath until the conversion of the metal into a white powder is complete. Then evaporate completely, stir the residue with 10 c.c. of nitric acid (sp.gr. 1.150 to 1.152) and 50 c.c. of water, and filter. To the filtrate add 1 c.c. of diluted sulphuric acid, evaporate on the water-bath as far as possible, and then take up the residue with 10 c.c. of water. In case the solution is not perfectly clear, filter. The weight of the insoluble matter after ignition should not exceed 0.0005 gm. ($PbSO_4$). (Indicating not more than 0.00683 per cent Pb.) To the filtrate add ammonia water to alkaline reaction. The liquid should not acquire a blue color. (Indicating less than 0.0004 per cent Cu.) Now add ammonium sulphide solution, and allow the mixture to stand for four to five hours at about 50° C. Should any precipitate form, its weight after ignition should not exceed 0.002 gm. (Indicating not over 0.04 per cent Fe and Zn as sulphides.)

Antimony. — Heat on the water-bath 5 gm. of tin and 50 c.c. of hydrochloric acid, adding potassium chlorate in small portions until solution results. Now heat the solution nearly to boiling and add bright piano-wire until the latter ceases to dissolve. (The piano-wire used in this test should be soluble without residue in dilute hydrochloric acid.) From this treatment no precipitate should result, or only a very slight black precipitate. (Indicating at most a trace.)

Arsenic. — Digest 5 gm. of tin on the water-bath with 40 c.c. of nitric acid (sp.gr. 1.4) until the metal is completely changed to a white powder. Evaporate completely, treat the residue with 10 c.c. of nitric acid (sp.gr. 1.150 to 1.152) and 50 c.c. of water, and filter. Mix the filtrate with 5 c.c. of concentrated sulphuric acid and evaporate, at first on a water-bath, then on a sand-bath, until fumes of sulphuric acid begin to be evolved. After cooling, dissolve the residue in 50 c.c. of water.

Start the evolution of hydrogen in a Marsh apparatus by means of 20 gm. of granulated arsenic-free zinc and diluted sulphuric acid (1 : 5), and gradually add the solution of the residue to the generating flask of the apparatus. After one hour there should be visible in the reduction tube at most a slight deposit of arsenic. (Indicating at most a trace.)

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TIN CHLORIDE

(Stannous Chloride)

$SnCl_2+2H_2O$. Mol. Wt. 225.95

Colorless crystals, easily and completely soluble in alcohol, and in water acidulated with hydrochloric acid. Stannous chloride solution is decomposed by much water, with the separation of a basic stannous chloride.

TESTS OF PURITY

Sulphates. — Dissolve 1 gm. of stannous chloride in 5 c.c. of hydrochloric acid (sp.gr. 1.19), and dilute with 50 c.c. of water. The solution should not be affected by barium chloride solution. (Indicating less than 0.125 per cent SO_3.)

Ammonium Compounds. — On heating 1 gm. of stannous chloride with 10 c.c. of sodium hydroxide solution (sp.gr. 1.3), no vapors of ammonia should be evolved (to be ascertained by moist litmus paper). (Indicating less than 0.0035 per cent NH_3.)

Earths, Alkalies, and Iron. — Dissolve 2 gm. of stannous chloride in 10 c.c. of hydrochloric acid (sp.gr. 1.19), dilute the solution with 100 c.c. of water, and pass into the solution hydrogen sulphide gas until all the tin has been precipitated. Filter off the precipitate, evaporate the filtrate, and gnite the residue; the weight of the ignited residue should not exceed 0.002 gm. (Indicating not more than 0.1 per cent earths and alkalies.) On heating the ignited residue with 1 c.c. of hydrochloric acid, then diluting with 20 c.c. of water, and adding potassium sulpho-cyanate solution, at most a slight red color should develop. (Indicating less than 0.00075 per cent Fe.)

Arsenic. — Boil 2 gm. of stannous chloride with 10 c.c. of hydrochloric acid (sp.gr. 1.19) for several minutes. The liquid should remain clear and colorless for one hour. (Indicating less than 0.00075 per cent As.)

Quantitative Determination. — Dissolve 0.5 gm. of stannous chloride in 2 c.c. of hydrochloric acid (sp.gr. 1.19) and dilute the solution with 50 c.c. of water. Then add 5 gm. of potassium and sodium tartrate, and sodium bicarbonate until the solution is alkaline to litmus paper. Now titrate with decinormal solution of iodine, using starch solution as the indicator.

1 c.c. of decinormal I = 0.0112976 gm. of $SnCl_2+2H_2O$, log. 05299.

TIN CHLORIDE SOLUTION

(Stannous Chloride Solution)

A slightly yellow, refractive liquid, the specific gravity of which should not be less than 1.90. It is obtained by stirring 5 parts of stannous chloride with 1 part of hydrochloric acid, and then saturating the mixture with dry hydrochloric acid gas.

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TESTS OF PURITY

Substances Precipitated by Alcohol. — On mixing stannous chloride solution with 10 times its volume of 85 per cent (by weight) alcohol, no turbidity should ensue on standing for one hour. (Indicating none present.)

Sulphuric Acid. — 5 c.c. of stannous chloride solution diluted with 50 c.c. of water should not become turbid within ten minutes after the addition of barium nitrate solution. (Indicating less than 0.002 per cent SO_3.)

URANIUM ACETATE, FREE FROM SODIUM

(Uranyl Acetate)

$UO_2(C_2H_3O_2)_2+2H_2O$. **Mol. Wt. 424.58**

A yellow, crystalline powder, easily soluble in water. As the preparation nearly always contains some basic salt, a clear solution is obtained only on adding a little acetic acid.

TESTS OF PURITY

Sulphates. — The solution of 1 gm. of uranium acetate in 20 c.c. of water and 2 to 3 c.c. of diluted acetic acid should not be affected by barium chloride solution. (Indicating less than 0.0035 per cent SO_3.)

Sodium. — Dissolve 5 gm. of uranium acetate in 200 c.c. of water, with the aid of 10 c.c. of diluted acetic acid. Add to the boiling solution an excess of ammonia water, filter, and evaporate the filtrate to dryness. Ignite the residue, dissolve it in water, and then titrate with normal hydrochloric acid solution, using methyl orange as the indicator. Not more than 0.1 c.c. of normal hydrochloric acid solution should be required to produce the red color. (Indicating not more than 0.046 per cent Na.)

Earths. — The solution of 1 gm. of uranium acetate in 20 c.c. of water and 2 to 3 c.c. of diluted acetic acid should not appear changed within one minute on adding ammonia water and ammonium carbonate solution in excess. (Indicating less than 0.005 per cent earths as Ca.)

Uranous Salt. — The solution of 1 gm. of uranium acetate in 20 c.c. of water and 1 c.c. of diluted sulphuric acid should be colored red on the addition of 0.1 to 0.2 c.c. of decinormal potassium permanganate solution. (Indicating not over 0.2385 per cent U^{IV}.)

Foreign Metals. — (*a*) On heating to boiling a solution of 5 gm. of uranium acetate in 100 c.c. of water and 5 c.c. of hydrochloric acid, no change should appear within one minute on passing into the solution hydrogen sulphide gas. (Indicating none present.)

(*b*) On adding to the liquid obtained under the test for earths, 2 or 3 drops of ammonium sulphide solution, no dark-brown color should develop, nor should a precipitate form. (Indicating none present.)

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URANIUM NITRATE

(Uranyl Nitrate)

$UO_2(NO_3)_2+6H_2O$. Mol. Wt. 502.62

Yellow crystals, having a greenish luster by reflected light, and efflorescing superficially in dry air. Uranium nitrate is easily soluble in water, alcohol, and ether. The aqueous solution is acid to litmus paper.

TESTS OF PURITY

Sulphates. — The 1 : 20 aqueous solution should not develop a turbidity within fifteen minutes on adding barium chloride solution. (Indicating less than 0.0025 per cent SO_3.)

Alkali Salts. — Ignite 1 gm. of uranium nitrate, pulverize the residue, ignite again, and treat the residue with 20 c.c. of water. On filtering, and evaporating the filtrate, no weighable residue should remain. (Indicating less than 0.05 per cent.)

Earths. — The solution of 1 gm. of uranium nitrate in 20 c.c. of water should not appear changed within one minute on the addition of ammonia water and ammonium carbonate solution in excess. (Indicating less than 0.005 per cent earths as Ca.)

Uranous Salt. — The solution of 1 gm. of uranium nitrate in 20 c.c. of water and 1 c.c. of diluted sulphuric acid should be colored red on the addition of 0.1 to 0.2 c.c. of decinormal potassium permanganate solution. (Indicating not more than 0.2385 per cent U^{IV}.)

Foreign Metals. — (*a*) The liquid obtained under the test for earths, when treated with 2 or 3 drops of ammonium sulphide solution, should not acquire a dark-brown color, nor should it develop a precipitate. (Indicating none present.)

(*b*) On heating to boiling a solution of 5 gm. of uranium nitrate in 100 c.c. of water and 5 c.c. of hydrochloric acid, and passing hydrogen sulphide gas into the solution, no change should appear. (Indicating none present.)

WATER, DISTILLED

H_2O. Mol. Wt. 18.02

Distilled water must be neutral to litmus paper.

TESTS OF PURITY

Ammonia and Ammonium Compounds. — 50 c.c. of the water should show no change on the addition of 10 to 15 drops of Nessler's reagent. (Indicating less than 0.00002 per cent NH_3.)

Chlorides. — 100 c.c. of water should show no change on adding a few drops of nitric acid followed by silver nitrate solution. (Indicating less than 0.00001 per cent Cl.)

Sulphates. — On adding 1 c.c. of hydrochloric acid and some barium chloride solution to 100 c.c. of water, no precipitate of barium sulphate should form on standing fifteen hours. (Indicating less than 0.0002 per cent SO_3.)

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Nitrates. — Introduce 5 c.c. of diphenylamine solution (see Diphenylamine) into a test-tube, and overlay it with 10 c.c. of water. No blue color should form at the contact-surfaces of the two liquids. (Indicating less than 0.0007 per cent N_2O_5.)

Non-volatile Matter. — 100 c.c. of the water evaporated on the water-bath should leave no weighable residue. (Indicating less than 0.0005 per cent.)

Heavy Metals and Calcium. — 100 c.c. of the water should show no change with hydrogen sulphide water (indicating no heavy metals present), or ammonia water with ammonium sulphide, or ammonium oxalate solution. (Indicating less than 0.0002 per cent Ca.)

Substances Oxidizable by Permanganate (Organic Matter, Nitrites, etc.). — Heat to boiling 100 c.c. of the water with 1 c.c. of 16 per cent of sulphuric acid, add 1 drop of potassium permanganate solution (1 : 1000), and maintain the boiling for three minutes. The liquid should not be decolorized. (Indicating none present.)

XYLIDINE

$C_6H_3(CH_3)_2(NH_2)$. Mol. Wt. 121.10

A yellow to yellowish-brown liquid, having a specific gravity of 0.981 to 0.984, and boiling at 212° to 215° C.

TEST OF PURITY

Substances Insoluble in Hydrochloric Acid. — 10 c.c. of xylidine should afford a perfectly clear solution with a mixture of 10 c.c. of hydrochloric acid and 10 c.c. of water. (Indicating none present.)

ZINC

Zn. Atomic Wt. 65.37

A shining, bluish-white metal.

I

ZINC, FREE FROM ARSENIC, SULPHUR, PHOSPHORUS, AND IRON

This grade of zinc occurs in three forms, granulated, thin sticks, and thick sticks.

TESTS OF PURITY

Arsenic. — Introduce 20 gm. of zinc into the generating flask of a Marsh apparatus, and start the hydrogen with arsenic-free, dilute sulphuric acid (1 : 5), maintaining the flow of gas until the metal is almost completely dissolved. At the end of the test, no deposit of arsenic should be visible within the reduction tube. (Indicating less than 0.000025 per cent As.)

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Matter Oxidizable by Potassium Permanganate. — Dissolve 10 gm. of zinc in a mixture of 60 c.c. of water and 15 c.c. of pure sulphuric acid (sp.gr. 1.84) in a flask provided with a rubber valve; no black flocks should remain undissolved. As soon as the zinc has dissolved, add to the solution, drop by drop, decinormal potassium permanganate solution. Not more than 0.1 c.c. should be required to produce a distinct pink color. Should more of the permanganate solution be required, the dilute sulphuric acid (15 c.c. of sulphuric acid and 60 c.c. of water) should be titrated for the purpose of control, without zinc, using the same solution of decinormal potassium permanganate. (Indicating less than 0.0056 per cent oxidizable matter as Fe.)

Compounds of Sulphur, Phosphorus, etc. — Into a narrow test-tube introduce 1 gm. of zinc together with 5 to 10 c.c. of dilute, arsenic-free sulphuric acid, and in the upper part of the tube insert a plug of cotton, which serves to hold back the water carried off by the escaping hydrogen. Over the mouth of the tube lay a small piece of filter paper which has been moistened with 1 : 1 silver nitrate solution, and carefully dried. The reaction is allowed to proceed in a dark place which is perfectly free from hydrogen sulphide. On standing two hours, the silver nitrate paper should show neither a yellow nor a black color. (Indicating none present.)

II

ZINC, FREE FROM ARSENIC, NEARLY FREE FROM IRON

This grade of zinc is in various forms, granulated, thick sticks, thin sticks, plates, powder, and turnings.

TESTS OF PURITY

Arsenic. — The test is carried out as described under Zinc, Free from Arsenic, Sulphur, Phosphorus, and Iron.

Matter Oxidizable by Permanganate. — Dissolve 10 gm. of zinc in a mixture of 60 c.c. of water and 15 c.c. of pure concentrated sulphuric acid in a flask provided with a rubber valve. The solution should contain only a very slight quantity of undissolved black flocks. As soon as the zinc has dissolved, add to the solution, drop by drop, decinormal potassium permanganate solution. Not more than 0.5 c.c. should be required to produce a distinct pink color. (Indicating less than 0.028 per cent oxidizable matter as Fe.)

This grade of zinc *in powder form* should, in addition to the above, be tested as follows:

Chlorides. — Dissolve 5 gm. of the zinc in a mixture of 50 c.c. of nitric acid and 100 c.c. of water. This solution should not show more than a slight opalescent turbidity on the addition of silver nitrate solution. (Indicating less than 0.0005 per cent Cl.)

III

ZINC, FREE FROM ARSENIC

This zinc is used in the following forms: granulated, thick sticks, thin sticks, powder and turnings.

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TEST OF PURITY

Arsenic. — This test is carried out as described under Zinc, Free from Arsenic, Sulphur, Phosphorus, and Iron.

IV

ZINC DUST

A fine, gray powder, containing about 90 per cent of metallic zinc.

TESTS OF PURITY

Nitrogen. — Dissolve 20 gm. of zinc dust in a mixture of 20 c.c. of sulphuric acid (sp.gr. 1.84) and 200 c.c. of water with the aid of heat. In case the zinc dust dissolves with great difficulty add a few drops of platinum chloride solution, free from nitric acid. After cooling, add to the solution 100 c.c. of sodium hydroxide solution I, distil off about 75 c.c. and collect the distillate in a receiver containing 10 c.c. of water and 2 to 3 c.c. of fifth-normal hydrochloric acid solution. Titrate the distillate with fifth-normal potassium hydroxide solution, using methyl orange as the indicator. Not more than 0.3 c.c. of the acid should have been neutralized by the ammonia. (Indicating not more than 0.00112 per cent N.)

Determining the Value of Zinc Dust. — Introduce 1 gm. of zinc dust together with a few glass beads into a flask of about 200 c.c. capacity, closed with a glass stopper, and then add a mixture of 30 c.c. of potassium iodate solution and 100 c.c. of sodium hydroxide solution (potassium iodate solution: 15.25 gm. KIO_3, to 300 c.c. H_2O; sodium hydroxide solution: 300 gm. NaOH to 1000 c.c. H_2O). Now shake the contents of the flask vigorously for five minutes; transfer, without filtering, to a graduated flask of 1000 c.c. capacity; fill with water up to the mark and mix. Of this solution transfer 100 c.c. into an iodine distillation apparatus. Add 50 c.c. of diluted sulphuric acid, and after expelling the air by means of carbon dioxide, distil, and receive the iodine in potassium iodide solution (1 : 5). The distillation is ordinarily complete in about twenty minutes, and may be recognized by the contents of the retort having become colorless. The iodine distilled over is titrated with decinormal sodium thiosulphate solution. $6I = 15Zn$.*

1 c.c. of decinormal $Na_2S_2O_3 = 0.0163425$ gm. of Zn, log. 21332.

ZINC CHLORIDE

$ZnCl_2$. Mol. Wt. 136.29

A white powder, deliquescent in the air, and easily soluble in water and in alcohol. The aqueous solution is acid to litmus paper.

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* $15Zn + 30NaOH = 15Zn(ONa)_2 + 15H_2$.
$5KIO_3 + 15H_2 = 5KI + 15H_2O$.
$KIO_3 + 5KI + 3H_2SO_4 = 3K_2SO_4 + 3H_2O + 6I$.

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TESTS OF PURITY

Zinc Oxychloride. — The solution of 1 gm. of zinc chloride in 1 c.c. of boiled water should be clear, or at most only slightly turbid; any turbidity caused in the solution by the addition of 3 c.c. of alcohol (85 per cent by weight) should disappear on the addition of 0.05 c.c. of hydrochloric acid. (Indicating less than 2.5 per cent as ZnO.)

Sulphates. — 10 c.c. of the 1 : 10 aqueous solution acidulated with a few drops of hydrochloric acid should not be rendered turbid by barium chloride solution. (Indicating less than 0.002 per cent SO_3.)

Foreign Metals and Alkalies. — (*a*) The solution of 1 gm. of zinc chloride in 10 c.c. of water acidulated with 1 c.c. of hydrochloric acid should appear unchanged after the addition of hydrogen sulphide water. (Indicating no foreign metals present.)

(*b*) The solution of 1 gm. of zinc chloride dissolved in 10 c.c. of water should remain clear and colorless on being mixed with 10 c.c. of ammonia water. On adding 5 drops of hydrogen sulphide water to the solution, a pure white precipitate should form. (Indicating no foreign metals present.) Precipitate the zinc completely by the further addition of hydrogen sulphide water. On filtering, evaporating the filtrate to dryness, and igniting, no weighable residue should remain. (Indicating less than 0.05 per cent alkali salts.)

ZINC IODIDE-STARCH SOLUTION

A colorless, clear, or only slightly opalescent liquid. It deteriorates with age, acquiring a blue color.

TESTS OF SENSITIVENESS

A mixture of 1 c.c. of zinc iodide-starch solution and 20 c.c. of water should acquire an intense blue color on adding 1 drop of decinormal iodine solution.

1 c.c. of zinc iodide-starch solution diluted with 50 c.c. of water should not acquire a blue color on the addition of diluted sulphuric acid.

ZINC OXIDE

ZnO. Mol. Wt. 81.37

A white, amorphous powder, having a faint yellowish tinge. Zinc oxide is insoluble in water, but is soluble in dilute acetic acid and in mineral acids.

TESTS OF PURITY

Arsenic. — A mixture of 1 gm. of zinc oxide and 3 c.c. of stannous chloride solution should not acquire a darker color on standing one hour. (Indicating less than 0.0015 per cent As.)

Sulphates and Chlorides. — On shaking 2 gm. of zinc oxide with 20 c.c. of water and filtering, the filtrate acidulated with nitric acid should show no reaction

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on the addition of barium nitrate solution, and should acquire at most a slight opalescent turbidity on the addition of silver nitrate solution. (Indicating less than 0.0075 per cent SO_3, and less than 0.001 per cent Cl.)

Nitrate. — Dissolve 1 gm. of zinc oxide in 10 c.c. of diluted acetic acid, and add 1 drop of 1 : 1000 indigo solution, a small crystal of sodium chloride, and 10 c.c. of concentrated sulphuric acid. The blue color of the liquid should not disappear on shaking. (Indicating less than 0.0016 per cent N_2O_5.)

Carbonates, Calcium, Magnesium, and Foreign Heavy Metals. — 1 gm. ot zinc oxide should dissolve completely in 10 c.c. of diluted acetic acid withouf effervescence. (Indicating less than 1 per cent CO_2.) On adding to this solution 20 c.c.of ammonia water, a clear, colorless liquid should result which should not show a change on the addition of ammonium oxalate and sodium phosphate solutions (indicating less than 0.02 per cent Ca and less than 0.005 per cent Mg), and which should give a pure white precipitate with 5 drops of hydrogen sulphide water. (Indicating no foreign heavy metals present.)

Foreign Bodies which Reduce Permanganate when Zinc Oxide is Dissolved in Diluted Sulphuric Acid. — Very carefully triturate 3 gm. of zinc oxide in a mortar with 20 c.c. of water containing in solution 0.2 gm. of pure ferric alum free from ferrous salt. Then add to the mixture 25 c.c.of diluted sulphuric acid, and effect complete solution by gently heating. Dilute the solution with 100 c.c. of thoroughly boiled and cooled water, and add decinormal potassium permanganate solution. One drop of the permanganate solution should suffice to impart a distinct pink color to the solution. (Indicating none present.)

ZINC SULPHATE

$ZnSO_4+7H_2O$. Mol. Wt. 287.55

Colorless crystals, efflorescent in dry air, soluble in 0.6 part of water, and almost insoluble in alcohol. The aqueous solution is acid to litmus paper.

TESTS OF PURITY

Chlorides. — The 1 : 20 aqueous solution should not be affected by silver nitrate solution. (Indicating less than 0.001 per cent Cl.)

Foreign Metals. — 0.5 gm. of zinc sulphate should dissolve in a mixture of 10 c.c. of water and 5 c.c. of ammonia water, affording a clear and colorless solution. This solution should afford a pure white precipitate with 5 drops of hydrogen sulphide water. (Indicating none present.)

Iron. — 1 gm. of zinc sulphate dissolved in 20 c.c. of water acidulated with a few drops of nitric acid and boiled should not be reddened by potassium sulphocyanate solution. (Indicating less than 0.0008 per cent Fe.)

Nitrates. — Add to 10 c.c. of a 1 : 10 zinc sulphate solution 1 drop of indigo solution, a small crystal of sodium chloride, and 10 c.c. of concentrated sulphuric acid. The blue color should not disappear on shaking. (Indicating less than 0.0016 per cent N_2O_5.)

Ammonium Compounds. — On heating 1 gm. of zinc sulphate with sodium hydroxide solution, no vapors of ammonia should be evolved (to be ascertained by moist litmus paper. (Indicating less than 0.0035 per cent NH_3.)

Free Sulphuric Acid. — On shaking 2 gm. of zinc sulphate with 10 c.c. of 85 per cent (by weight) alcohol, and, after about ten minutes, filtering, the

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filtrate, when diluted with 10 c.c. of water, should not redden blue litmus paper. (Indicating none present.)

Arsenic. — Introduce 20 gm. of arsenic-free, metallic zinc into the generating flask of a Marsh apparatus, and start the hydrogen with dilute (1 : 5) sulphuric acid. Dissolve 2 gm. of zinc sulphate in 20 c.c. of water, introduce the solution in small quantities at a time into the generating flask of the Marsh apparatus. After one hour no deposit of arsenic should be visible within the reduction tube. (Indicating less than 0.0005 per cent As.)

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1913

INTERNATIONAL ATOMIC WEIGHTS

O = 16

Aluminum	Al	27.1	Molybdenum	Mo	96.0
Antimony	Sb	120.2	Neodymium	Nd	144.3
Argon	A	39.88	Neon	Ne	20.2
Arsenic	As	74.96	Nickel	Ni	58.68
Barium	Ba	137.37	Niton (radium emanation)	Nt	222.4
Bismuth	Bi	208.0	Nitrogen	N	14.01
Boron	B	11.0	Osmium	Os	190.9
Bromine	Br	79.92	Oxygen	O	16.00
Cadmium	Cd	112.40	Palladium	Pd	106.7
Caesium	Cs	132.81	Phosphorus	P	31.04
Calcium	Ca	40.07	Platinum	Pt	195.2
Carbon	C	12.00	Potassium	K	39.10
Cerium	Ce	140.25	Praseodymium	Pr	140.6
Chlorine	Cl	35.46	Radium	Rd	226.4
Chromium	Cr	52.0	Rhodium	Rh	102.9
Cobalt	Co	58.97	Rubidium	Rb	85.45
Columbium	Cb	93.5	Ruthenium	Ru	101.7
Copper	Cu	63.57	Samarium	Sm	150.4
Dysprosium	Dy	162.5	Scandium	Sc	44.1
Erbium	Er	167.7	Selenium	Se	79.2
Europium	Eu	152.0	Silicon	Si	28.3
Fluorine	F	19.0	Silver	Ag	107.88
Gadolinium	Gd	157.3	Sodium	Na	23.00
Gallium	Ga	69.9	Strontium	Sr	87.63
Germanium	Ge	72.5	Sulphur	S	32.07
Glucinum	Gl	9.1	Tantalum	Ta	181.5
Gold	Au	197.2	Tellurium	Te	127.5
Helium	He	3.99	Terbium	Tb	159.2
Holmium	Ho	163.5	Thallium	Tl	204.0
Hydrogen	H	1.008	Thorium	Th	232.4
Indium	In	114.8	Thulium	Tm	168.5
Iodine	I	126.92	Tin	Sn	119.0
Iridium	Ir	193.1	Titanium	Ti	48.1
Iron	Fe	55.84	Tungsten	W	184.0
Krypton	Kr	82.92	Uranium	U	238.5
Lanthanum	La	139.0	Vanadium	V	51.0
Lead	Pb	207.10	Xenon	Xe	130.2
Lithium	Li	6.94	Ytterbium (Neoytterbium)	Yb	172.0
Lutecium	Lu	174.00	Yttrium	Y	89.0
Magnesium	Mg	24.32	Zinc	Zn	65.37
Manganese	Mn	54.93	Zirconium	Zr	90.6
Mercury	Hg	200.6			

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PREPARATION OF TEST SOLUTIONS COMMONLY USED

Test Solution.	Approx. Percentage Strength Generally Used.	Quantity of Chemical Dissolved in Water to Make 250 c.c.	Approx. Normality Generally Used.	Quantity of Chemical Dissolved in Water to Make 250 c.c.
Acid Acetic	36 %	90 gm. (of 99.5 % acid)	5 N	75 gm. (of 99.5% acid)
Hydrobromic	10%	67 gm. (of 40% acid)	0.5 N	25gm. (of 40% acid)
Hydriodic	10%	60 gm. (of acid sp.gr. 1.5)	0.5 N	37 gm. (of acid sp.gr. 1.5)
Hydrochloric	31.9%	255 gm. (of acid sp.gr. 1.19)	5 N	120 gm. (of acid sp.gr. 1.19)
Hydrosilicofluoric	7.5%	Acid Hydrosilicofluoric (undiluted)		
Iodic	1.6%	4 gm.	0.5 N	4 gm.
Nitric	10%	38 gm. (of acid sp.gr. 1.40–1.42)	5 N	113 gm. (of acid sp.gr. 1.40–1.42)
Nitro-hydrochloric		62 c.c. Acid Nitric sp. gr. 1.40–1.42 with 188 c.c. Acid Hydrochloric sp.gr. 1.19		
Oxalic	10%	25 gm.	1.58 N	25 gm.
Phosphoric	10%	31 gm. (of acid sp.gr. 1.7)	0.5 N	5 gm. (of acid sp. gr. 1.7)
Picric	1%	2.5 gm.	0.043 N	2.5 gm.
Sulphuric	95%	Acid Sulphuric sp.gr. 1.84 (undiluted)		
Sulphuric	10%	28 gm. (of acid sp.gr. 1.84)	5 N	64 gm. (of acid sp.gr. 1.84)
Sulphurous	6%	Acid Sulphurous (undiluted)		
Tartaric	25%	70 gm.	3.7 N	70 gm.
Ammon. Carbonate	20%	50 gm. (with 50 c.c. Ammonia Water, 10%)		
Chloride	10%	25 gm.	5 N	67 gm.
Hydroxide	10% (NH_3)	90 gm. (of 28%)	5 N	75 gm. (of Ammonia water, 28%)
Molybdate		18.75 gm. Ammonium Molybdate in 125 c.c. Ammonia Water, 10% Pour this into 125 c.c. Acid Nitric, sp.gr. 1.2.		
Oxalate	4%	10 gm.	0.5 N	10 gm.
Sulphate	10%	25 gm.	0.5 N	8 gm.
Sulphide		Ammonium Sulphide solution (undiluted)		
Polysulphide		Allow the colorless ammonium sulphide solution to stand for some time, or dissolve in it a small amount of pure sulphur.		
Arsenous Oxide		Saturated solution	0.25 N	2 gm.
Barium Chloride	10%	25 gm.	0.5 N	15 gm.
Hydroxide	Saturated solution	Barium Hydroxide Solution	0.2 N	32 gm.
Nitrate	10%	25 gm.	0.5 N	16 gm.
Calcium Chloride	10%	25 gm. (of crystals)	0.5 N	14 gm.
Hydroxide	0.14% (saturated)	0.35 gm. (Lime Water undiluted)	0.0378 N	0.35 gm.
Sulphate		Saturated solution		
Cobaltous Nitrate	10%	25 gm.	0.5 N	18 gm.
Cupric Chloride	4.4%	11 gm.	0.5 N	11 gm.

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PREPARATION OF TEST SOLUTIONS COMMONLY USED—*Continued*

Test Solution.	Approx. Percentage Strength Generally Used.	Quantity of Chemical Dissolved in Water to Make 250 c.c.	Approx. Normality Generally Used.	Quantity of Chemical Dissolved in Water to Make 250 c.c.
Cuprous Chloride	2.4%	6 gm.	0.5 N	6 gm.
Ferric Chloride	10%	25 gm.	0.5 N	7 gm.
Ferrous Sulphate	6.8%	17.5 gm. (with a few drops H_2SO_4). (Unstable.)	0.5 N	20 gm.
Hydrogen Peroxide	3%	25 gm. (of 30%)	0.5 N	7 gm. (of 30%)
Lead Acetate	10%	25 gm.		
Magnesia Mixture	(1 c.c. = 0.01 gm. P)	Magnesium Sulphate, 25 gm. Ammonium Chloride 50 gm. Ammonia Water 10%, 105 c.c. Water, 200 c.c.		
Magnesium Chloride	5.2%	13 gm.	0.5 N	13 gm.
Sulphate	10%	25 gm.	0.5 N	16 gm.
Manganous Chloride	4.8%	12 gm.	0.5 N	12.5 gm.
Sulphate	6.8%	17 gm.	0.5 N	17 gm.
Mercuric Chloride	5%	12.5 gm.	0.5 N	
Nitrate	60% (with 11% free acid)	150 gm. Mercuric Nitrate, 40 gm. Acid Nitric (sp.gr.1.40-142)	0.5 N	20 gm.
Palladous Sodium Chloride	8.4%	21 gm.	0.57 N	21 gm.
Potassium Bromate	1.4%	3.5 gm.	0.5 N	3.5 gm.
Bromide	6%	15 gm.	0.5 N	15 gm.
Carbonate	10%	25 gm.	3 N	52 gm.
Chloride	3.6%	9 gm.	0.5 N	9 gm.
Chromate	10%	25 gm.	0.5 N	12 gm.
Dichromate	10%	25 gm.	0.5 N	6 gm.
Ferrocyanide	10%	25 gm.	0.5 N	13 gm.
Ferricyanide	10%	25 gm.	0.5 N	14 gm.
Hydroxide	5%	15 gm.	5 N	83 gm.
Iodate	1.78%	4.46 gm.	0.5 N	4.5 gm.
Iodide	20%	50 gm.	0.5 N	21 gm.
Nitrate	5.2%	13 gm.	0.5 N	13 gm.
Permanganate	0.32% (0.1 N)	0.8 gm.	0.5 N	4 gm.
Thiocyanate	1% (0.1 N)	2.5 gm.	0.5 N	12 gm.
Sulphate	1%	2.5 gm.	0.5 N	11 gm.
Platinic Chloride	13%	33 gm.		
Silver Nitrate	1.6% (0.1 N)	4 gm.	0.25 N	11 gm.
Sodium Acetate	10%	25 gm.	0.73 N	25 gm.
Carbonate	16%	40 gm. (of anhydrous salt)	3 N	40 gm.
Chloride	2.8%	7 gm.	0.5 N	7 gm.
Hydroxide	5%	14 gm.	5 N	55 gm.
Nitrate	4.4%	11 gm.	0.5 N	11 gm.
Phosphate	10%	25 gm.	0.5 N	15 gm.
Sulphate	3.6%	9 gm.	0.5 N	9 gm.
Sulphide	5% (Na_2S)	40 gm. (Na_2S+9H_2O)	0.5 N	15 gm. (Na_2S+9H_2O)
Sulphite	6.4%	16 gm.	0.5 N	16 gm.
Thiosulphate	2.4% (0.1 N)	6 gm.	0.1 N	6 gm.
Stannous Chloride	5.6%	14 gm.	0.5 N	14 gm.
Zinc Chloride	3.4%	8.5 gm.	0.5 N	8.5 gm.

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